

Research Article

Study on the Effect of Nanoindentation Test Method on Micromechanical Properties of Granite Minerals

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The study on microscopic mechanical properties of mineral is of great significance to reveal the damage mechanism of rock and can provide important basis for nondestructive testing of building materials and assisted rock breaking technology. As the main test method for rock and mineral microscopic mechanical properties, nanoindentation test has several parameters such as loading time, indentation depth, and loading rate, which have great influence on the test results. Three schemes are designed as follows: (1) The loading and unloading rates remain unchanged, and the holding time is 0 s, 5 s, 10 s, 20 s, 50 s, and 100 s, respectively. (2) Ten cycles of loading and unloading tests are used to calculate micromechanical indexes at different indentation depth. (3) The maximum load and holding time remain unchanged, and the loading rate is 5 mN/s, 10 mN/s, 20 mN/s, 30 mN/s, 40 mN/s, and 50 mN/s, respectively. The results show that (1) with the increase of holding time, the elastic modulus and hardness of feldspar decrease sharply between 0 s and 5 s, decrease slightly between 5 s and 10 s, and stabilize after 10 s. The creep displacement of feldspar is positively correlated with holding time. The holding time does not change the development path of creep displacement. (2) With the increase of indentation depth, the elastic modulus and hardness of feldspar decrease gradually and become stable when the indentation depth is more than 350 nm. (3) The elastic modulus and hardness of quartz increase with loading rate. The maximum indentation depth and residual indentation depth decrease with the increase of loading rate. The creep displacement increases with loading rate, while the creep compliance decreases with the increase of loading rate. With the increase of holding time, the creep rate decreases and tends to be stable. The results provide theoretical basis for obtaining more accurate rock nanoindentation mechanical properties and revealing rock damage mechanism.

1. Introduction

There are many ways to test the mechanical properties of rock. The conventional tensile and compression tests are destructive for sample. These experiments have strict requirements on sample size, shape, and quantity. It enhances on-site sampling requirements for engineers and technicians and increases the project cost. And in particular, these tests are useless for nondestructive testing of the completed projects. However, nanoindentation test is a repeatable technique with less sampling to detect local mechanical properties of material.

In recent years, nanoindentation technology has been introduced into the study of rock mechanical properties.

The determination of basic mechanical properties such as elastic modulus, hardness, creep index, and fracture toughness of minerals and their relationship are the focus of current research. Zhu et al. [1] used nanoindentation technology to draw rock nanomechanical properties maps. Liu et al. [2] studied the microscopic mechanical properties of shale based on nanoindentation technology. Zeng et al. [3] used an improved nanoindentation technology to measure the microscopic damage properties of shale. Sun et al. [4] studied the rheological properties of sandstone minerals by using nanoindentation technology. Zhang et al. [5] applied nanoindentation test to study the microscopic mechanical properties of granite, providing a reference for determining the macroscopic mechanical properties of rock

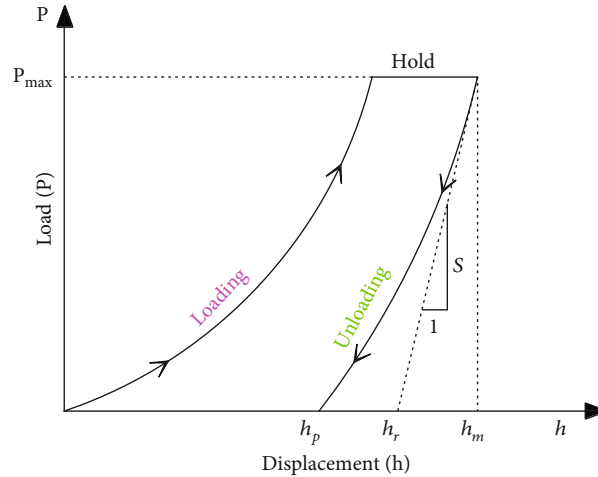


FIGURE 1: Typical nanoindentation load-displacement curve. h_p : residual indentation depth; h_r : intersection point between tangent line at the maximum load on unloading curve and displacement axis; h_m : maximum indentation depth.



FIGURE 2: Nano Indenter G200.

from a microscopic perspective. Liu et al. [6] obtained the quantitative relationship between fracture toughness and elastic modulus of shale based on energy analysis method using nanoindentation, which provides a good perspective for understanding the nanoscale behavior of rock.

There are many factors that affect the accuracy of nanoindentation test, such as determination of contact zero, indenter area function, thermal drift, and sample surface roughness [7]. Furthermore, the stress relaxation and material hardening caused by creep during holding process are closely related to holding time. The appropriate selection of holding time is very important to eliminate the effect of viscoelastic interaction between sample and indenter. Moreover, the elastic modulus and hardness of mineral are not constant along the indentation depth direction in nanoindentation test. The elastic modulus and hardness of material are large when indentation depth is small, and the two gradually tend to be constant with the increase of indentation depth [4, 8]. Last but not least, the difference of loading rate will cause the change of contact area, which will lead to the change of contact hardness [9]. Therefore, the reliability of nanoindentation test results is affected by holding time, indentation depth, and loading rate.

Taking feldspar and quartz in granite for example, the nanomechanical properties of minerals are measured under different holding time, indentation depth, and loading rate in nanoindentation test. The variation laws of mineral mechanical properties under different conditions are obtained, which provides a theoretical basis for revealing the damage mechanism of granite.

2. Test

2.1. Basic Principles of Nanoindentation. Figure 1 shows the three-stage process curves of typical sample under single loading, holding, and unloading [1]. In the loading stage, the load increases with indentation depth. This stage includes elastic deformation and plastic deformation. In the unloading stage, elastic deformation can be recovered, so this stage can be used to obtain the mechanical indexes such as elastic modulus and hardness of materials [4, 10].

Elastic modulus E , hardness H , creep index, and fracture toughness can be obtained by using nanoindentation test. Contact stiffness S is introduced to calculate elastic modulus and hardness and is defined as the slope of tangent line at the maximum load on the unloading curve, which can be

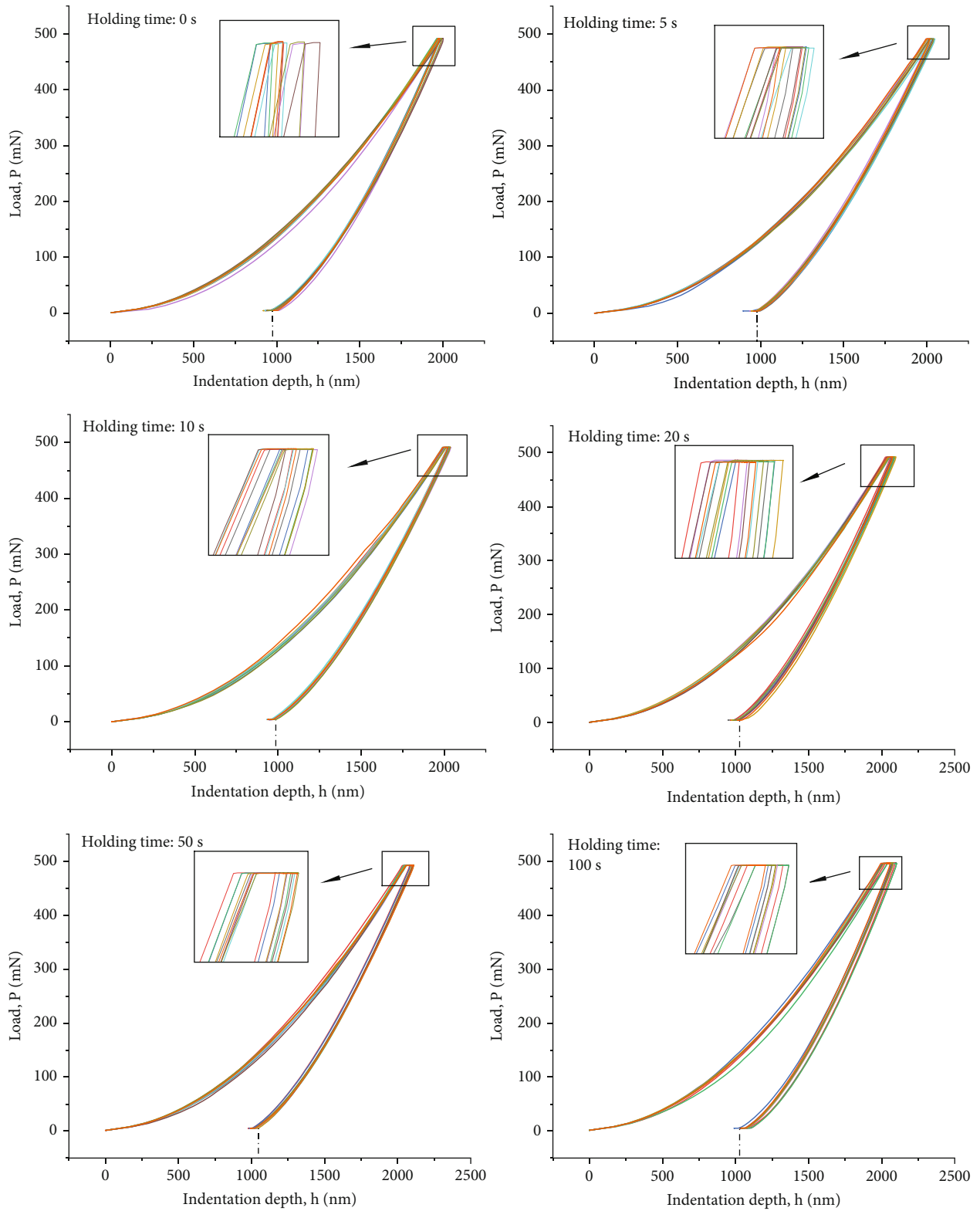


FIGURE 3: Load-displacement curves under different holding time conditions.

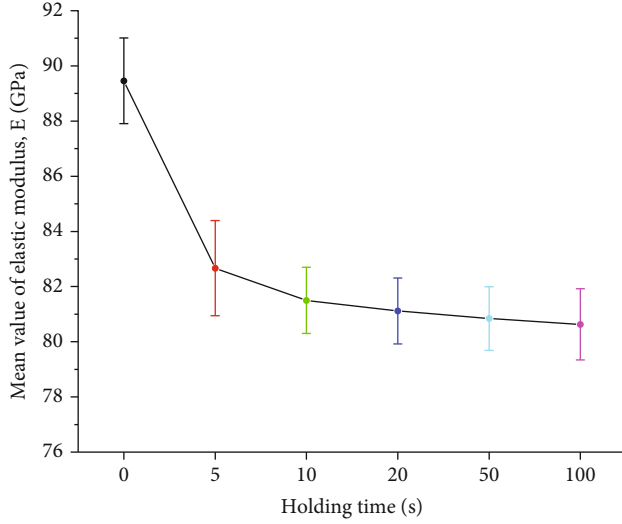


FIGURE 4: Variation of elastic modulus with holding time for feldspar.

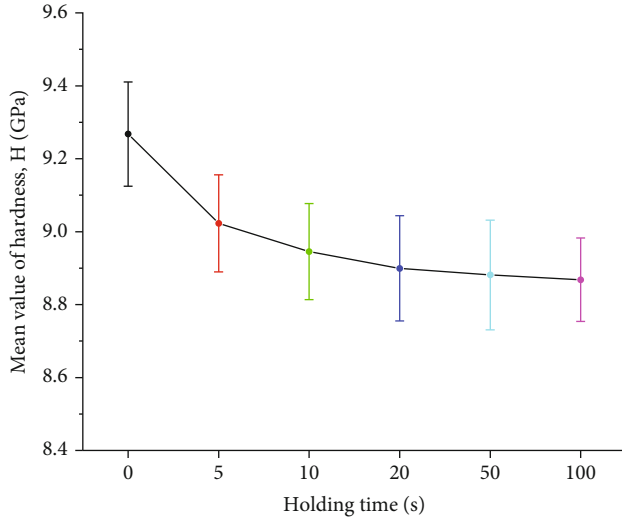


FIGURE 5: Variation of hardness with holding time for feldspar.

expressed as follows:

$$S = \frac{dP}{dh} \Big|_{h=h_m} \quad (1)$$

The reduced modulus is calculated as follows [11]:

$$E_r = \frac{\sqrt{\pi}}{2\beta} \cdot \frac{S}{\sqrt{A}} \quad (2)$$

The contact area A needs to be determined to calculate the reduced modulus and is related to contact depth h_c , which can be expressed as follows [12]:

$$A = 24.56h_c^2 \quad (3)$$

For the Berkovich indenter, the contact depth can be cal-

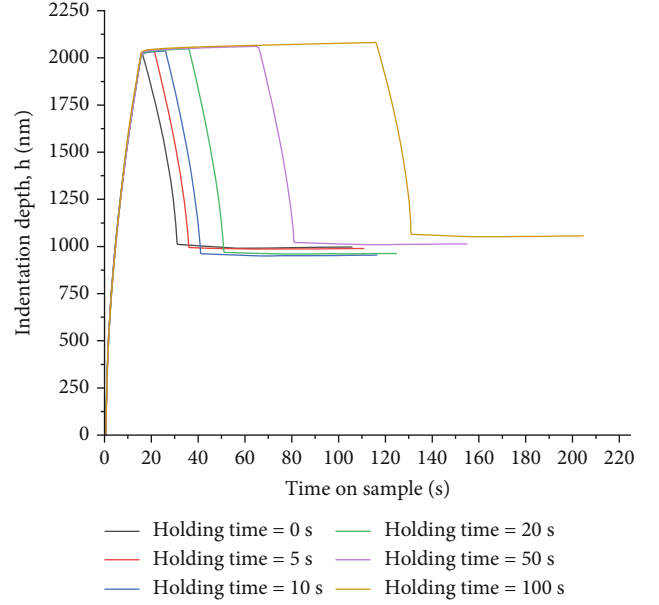


FIGURE 6: Variation of indentation depth with time for feldspar.

culated as follows:

$$h_c = h - \varepsilon \frac{P}{S} \quad (4)$$

The hardness is expressed as follows:

$$H = \frac{P}{A} \quad (5)$$

Elastic modulus can be calculated as follows:

$$\frac{1}{E_r} = \frac{(1 - \nu^2)}{E} + \frac{(1 - \nu_i^2)}{E_i} \quad (6)$$

where β and ε are constant dependent on the indenter shape. Ψ is the angle between the edge surface and the centerline. In this test, a modified Berkovich indenter with a regular triangular pyramid is used, $\Psi = 65.3^\circ$, $\beta = 1.034$, $\varepsilon = 0.75$. E_r is the reduced elastic modulus. A (nm^2) is the contact area. h_c (nm) is the contact depth. E_i and ν_i represent the elastic modulus and Poisson's ratio of indenter, respectively. For the diamond indenter, $E_i = 1141$ GPa, and $\nu_i = 0.07$. ν is the Poisson's ratio of sample.

2.2. Testing Device and Method. The nanoindentation test is carried out on the Nano Indenter G200, which is shown in Figure 2. The maximum load is 500 mN, and the load resolution is 50 nN. The Berkovich indenter is selected for this test. The maximum indenter displacement is 1.5 mm, the maximum indentation depth is 500 μm , and the displacement resolution is 0.01 nm.

- (1) With the same loading rate (30 mN/s) and the same maximum load (500 mN), the nanoindentation test of feldspar in granite is carried out when the holding

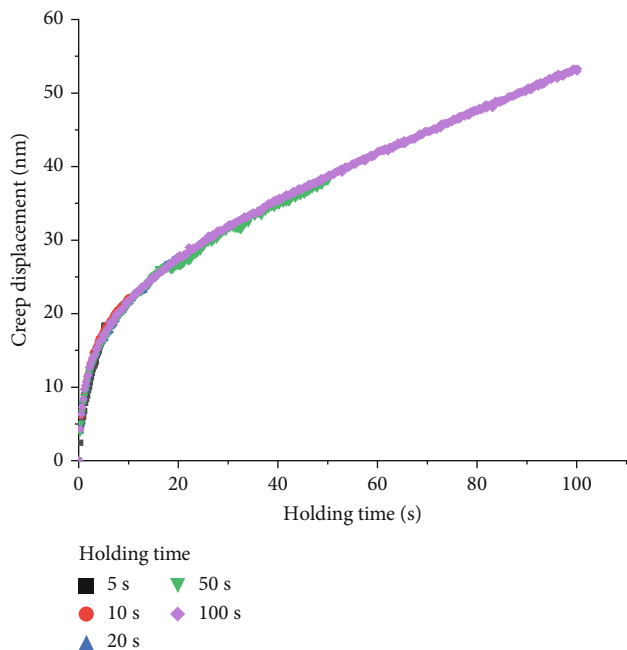


FIGURE 7: Variation of creep displacement with holding time for feldspar.

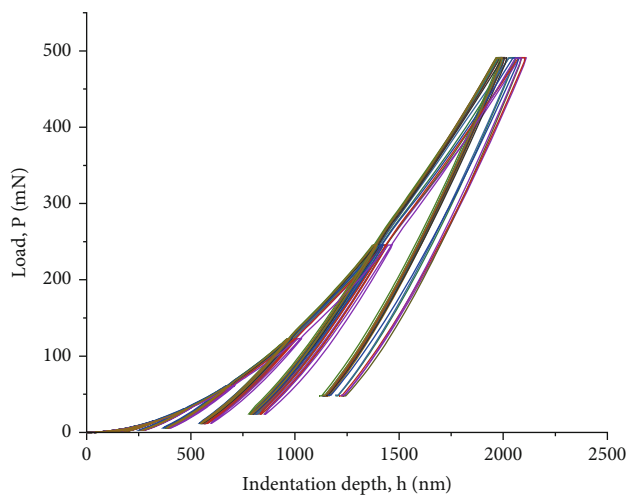


FIGURE 8: Load-displacement curve of feldspar under ten cyclic loading and unloading.

time is 0 s, 5 s, 10 s, 20 s, 50 s, and 100 s, respectively. The effects of holding time on nanomechanical indexes are studied

- (2) Nanoindentation test of feldspar in granite is carried out with a maximum load of 500 mN under ten cyclic loading and unloading. The effects of indentation depth on nanomechanical indexes are studied
- (3) With the same holding time (10 s) and the same maximum load (500 mN), the nanoindentation test of quartz in granite is carried out when the loading rate is 5 mN/s, 10 mN/s, 20 mN/s, 30 mN/s, 40 mN/s, and 50 mN/s, respectively. The effects of loading rate on nanomechanical indexes are studied

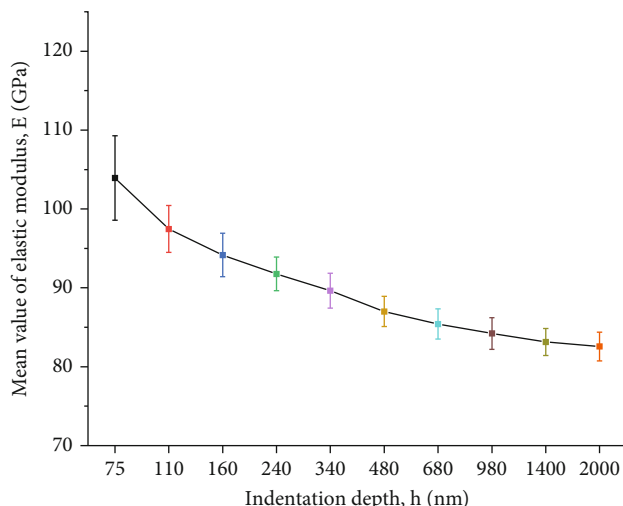


FIGURE 9: Variation of elastic modulus with indentation depth for feldspar.

3. Test Results and Analysis

3.1. Effect of Holding Time on Nanomechanical Properties. The holding time is time that the load keeps unchanged after the load increases to a certain degree in nanoindentation test. The material viscoelastic property reflects bond between indenter and specimen in nanoindentation test. Under this action, the indentation displacement increases with time under constant load; that is, the specimen creep occurs.

Under conditions that the maximum load is 500 mN and the holding time is 0 s, 5 s, 10 s, 20 s, 50 s, and 100 s, respectively, the load-displacement curves are shown in Figure 3. It can be seen that the longer the holding time is, the greater the creep displacement is and the deeper the residual indentation is.

Figure 4 shows the variation of elastic modulus with holding time for feldspar. It can be seen that the elastic modulus is the largest when the holding time is 0 s. With the increase of holding time, the elastic modulus of feldspar decreases sharply between 0 s and 5 s, decreases slightly between 5 s and 10 s, and stabilizes after 10 s. This is because that the apparent plastic deformation of feldspar occurs during the holding period. And holes, dislocations, and other defects in feldspar result in energy release. Then, the strengthening effect of feldspar is reduced, which contribute to stress relaxation. Finally, stress relaxation leads to elastic modulus reduction. Therefore, the energy liberation and elastic modulus gradually tend to be stable with the increase of the holding time.

Figure 5 shows the variation of hardness with holding time for feldspar. It can be seen that the hardness is the largest when the holding time is 0 s. With the increase of holding time, the hardness of feldspar decreases sharply between 0 s and 5 s, decreases slightly between 5 s and 10 s, and stabilizes after 10 s.

Figure 6 shows the variation of indentation depth with time for feldspar. Figure 7 shows the variation of creep displacement with holding time for feldspar. It can be seen that

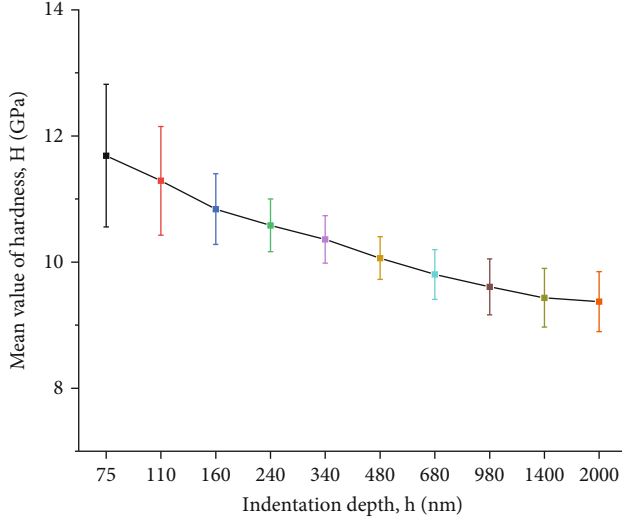


FIGURE 10: Variation of hardness with indentation depth for feldspar.

TABLE 1: Comparison of elastic modulus and hardness under different indentation depth.

Indentation depth (h/nm)	Elastic modulus (E/GPa)	Hardness (H/GPa)
75	103.92 ± 5.21	11.69 ± 1.10
110	97.47 ± 2.90	11.29 ± 0.84
160	94.17 ± 2.68	10.84 ± 0.55
240	91.77 ± 2.07	10.58 ± 0.41
340	89.63 ± 2.16	10.36 ± 0.37
480	87.00 ± 1.86	10.06 ± 0.33
680	85.42 ± 1.87	9.80 ± 0.38
980	84.21 ± 1.97	9.61 ± 0.43
1400	83.14 ± 1.67	9.43 ± 0.45
2000	82.57 ± 1.78	9.37 ± 0.46

the creep curves of feldspar during the same holding period coincide basically. The longer the holding time is, the greater the creep displacement is. The creep rate increases sharply at the initial stage of holding, then decreases gradually between 5 s and 30 s, and remains unchanged after 30 s. This indicates that the holding time does not change the development path of creep displacement.

3.2. Effect of Indentation Depth on Nanomechanical Properties. Figure 8 shows the load-displacement curve of feldspar under ten cyclic loading and unloading. It can be seen that the loading and unloading curves basically coincide without forming an obvious hysteresis loop. Figures 9 and 10 show the variation of elastic modulus and hardness with indentation depth for feldspar, respectively. The elastic modulus and hardness of feldspar decrease and tend to be stable with the increase of indentation depth. The comparison of elastic modulus and hardness under different indentation depth is shown in Table 1. It can be found that the indenta-

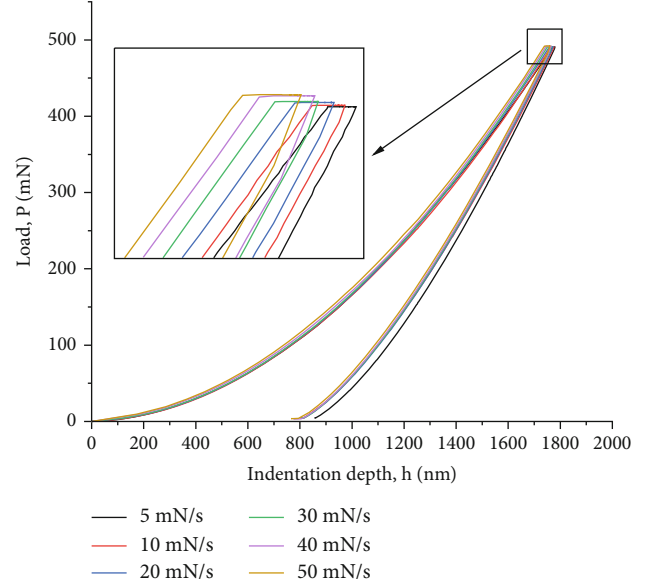


FIGURE 11: Load-displacement curves at different loading rates for quartz.

tion size effect appears in the experiment [13]. That is, when the load is very small, the elastic modulus and hardness of the material are very large, which are not real material parameters; the calculation of hardness not conforms to Mick's law within the small load range [14]. The results are influenced by many factors: (1) Affected by the vibration of the test system, vibration under small load will have a great influence on the results. (2) During the sample preparation process, the sample surface is hardened when polished. (3) Interparticle dislocation slip occurs when the load is small, and the deformation increases with load. The dislocation slip is limited when the load reaches a certain value. Therefore, the elastic modulus and hardness gradually tend to be stable with the increase of the load.

Therefore, it is necessary to select the appropriate load according to the material hardness in nanoindentation test. Under normal circumstances, small load should be used for soft material, and large load should be used for hard material. In addition, the hardness should not be expressed by a single value, but by a curve is more reasonable.

3.3. Effect of Loading Rate on Nanomechanical Properties. Figure 11 shows the load-displacement curves at different loading rates for quartz. The loading rate is 5 mN/s, 10 mN/s, 20 mN/s, 30 mN/s, 40 mN/s, and 50 mN/s, respectively. Figure 12 shows the variation of elastic modulus and hardness with loading rate for quartz. Figure 13 shows the variation of maximum indentation depth and residual indentation depth with loading rate for quartz. It can be seen that the elastic modulus and hardness increase with loading rate, while the maximum indentation depth and residual indentation depth decrease with the increase of loading rate. This is consistent with the existing researches [9, 15–17].

Creep compliance is a material parameter that reflects creep properties, which is the ratio of strain to stress at any time during creep. Creep compliance in uniaxial creep test

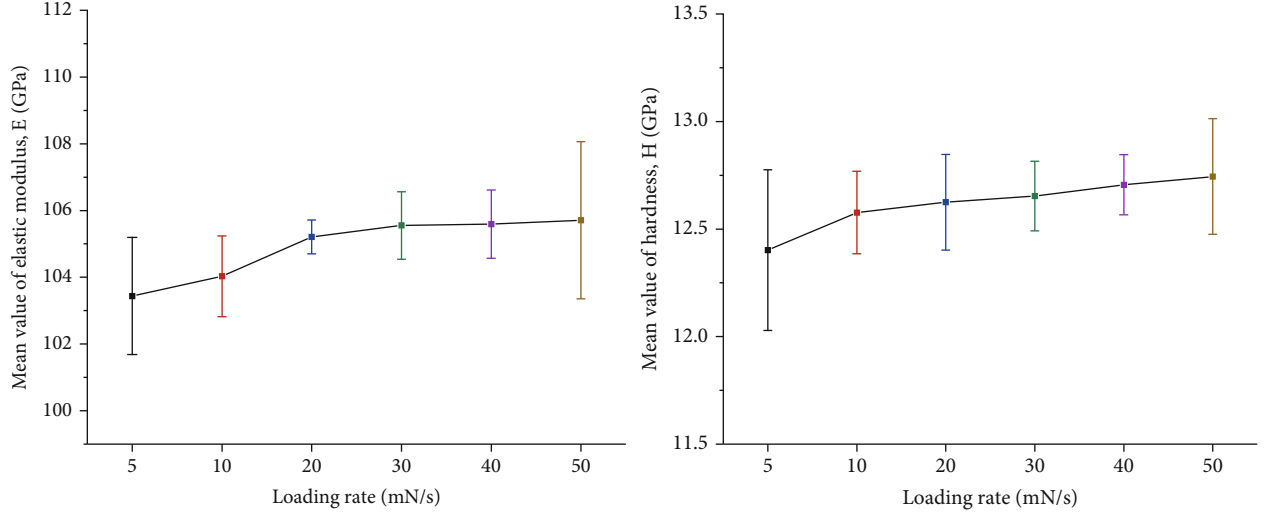


FIGURE 12: Variation of elastic modulus and hardness with loading rate for quartz.

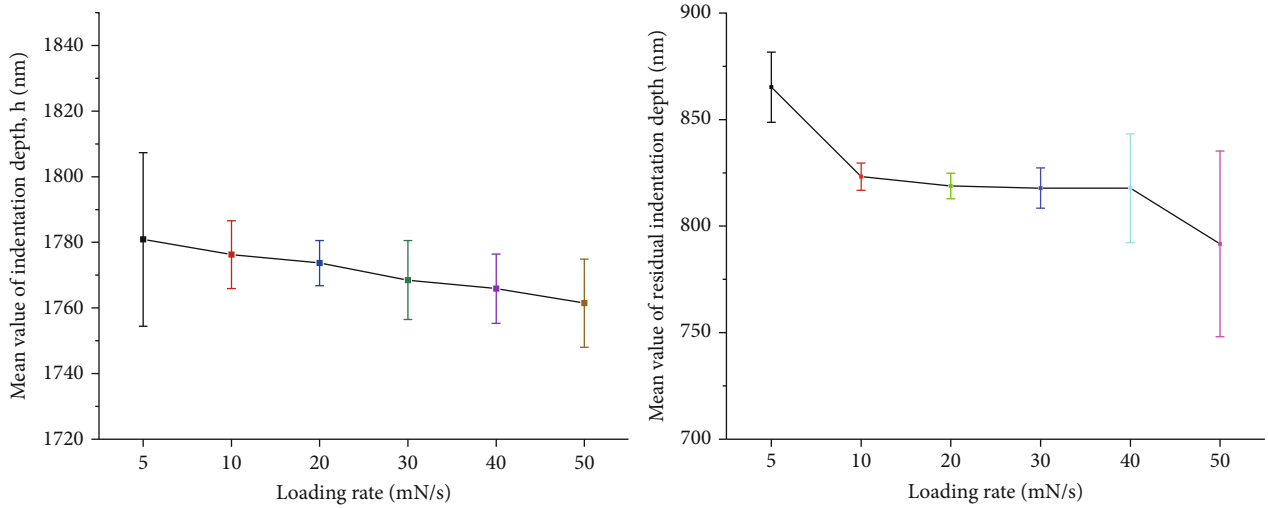


FIGURE 13: Variation of maximum indentation depth and residual indentation depth with loading rate for quartz.

is expressed as follows:

$$J(t) = \frac{\varepsilon(t)}{\sigma}, \quad (7)$$

where $J(t)$ is the creep compliance, $\varepsilon(t)$ is the strain during creep, and σ is the stress at any time. In the nanoindentation test, the creep compliance can be expressed as follows [18]:

$$J(t) = \frac{4h^2(t)}{\pi(1-\nu)P_{\max} \tan \alpha}, \quad (8)$$

where h is the indentation depth. P_{\max} is the load during holding time. The Berkovich indenter is modeled as a conical indenter with a half-cone angle of 70.3° , or $\alpha = 19.7^\circ$. ν is the Poisson's ratio of sample, for quartz, $\nu = 0.2$.

Figure 14 shows the variation of creep characteristic quantity with time at different loading rates for quartz when the holding time is 10 s. Figure 15 shows the variation of creep displacement with loading rate for quartz. As can be seen from Figures 14 and 15, creep displacement and the creep compliance increase with holding time. The creep displacement during the holding period increases with loading rate, while the creep compliance decreases with the increase of loading rate. In the initial stage of creep, the creep rate decreases sharply with holding time. This stage is called transient creep. Moreover, the larger the loading rate is, the larger the creep rate is. Due to the large loading rate, the plastic deformation has no time to release during the loading process and is released during the holding period. Conversely, when the loading rate is small, part of the plastic deformation has been released during the loading process. With the increase of holding time, the creep rate decreases and tends to be stable.

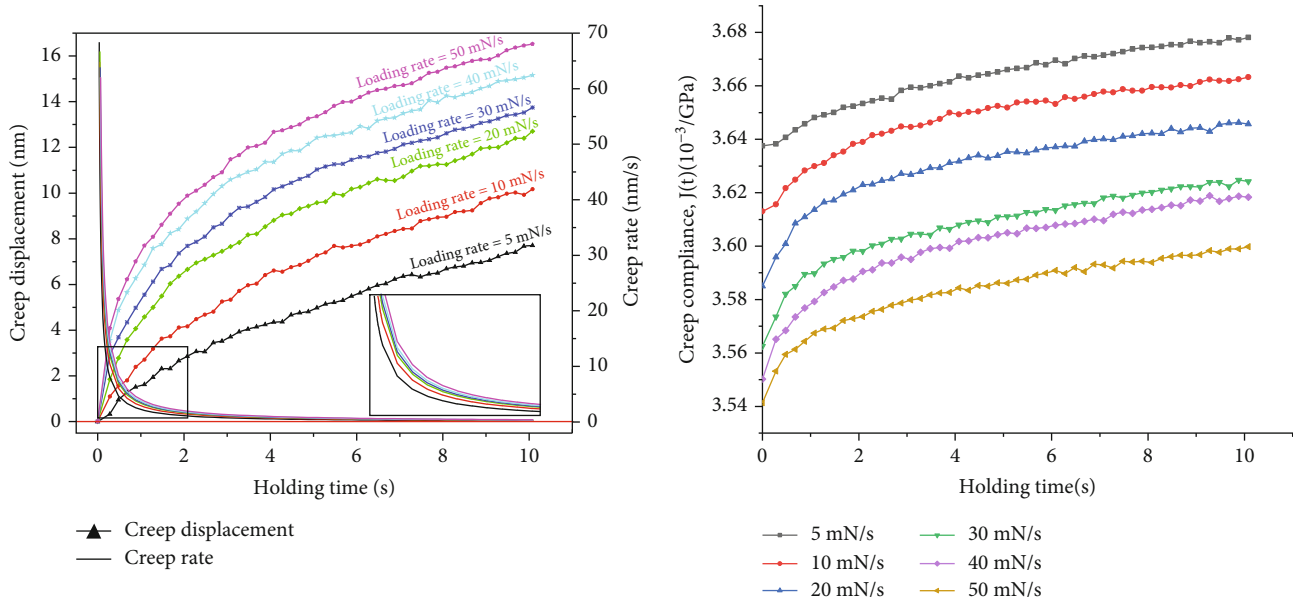


FIGURE 14: Variation of creep characteristic quantity with time at different loading rates for quartz.

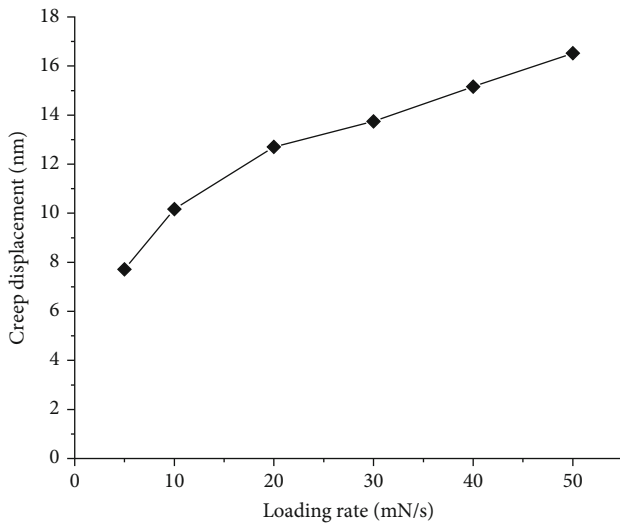


FIGURE 15: Variation of creep displacement with loading rate for quartz.

4. Conclusion

Taking quartz and feldspar as examples, the effects of holding time, indentation depth, and loading rate are studied on nanoindentation test. The conclusions are as follows:

- (1) Under the same loading rate and maximum load conditions, the elastic modulus and hardness of feldspar decrease sharply first, then decrease slightly, and become stable with holding time. The creep displacement increases with holding time, while the creep rate decreases with holding time. The holding time does not change the development path of creep displacement. That is, the creep curves of feldspar during the same holding period coincide basically

- (2) Through ten cyclic loading and unloading tests, micromechanical indexes at different indentation depth can be obtained. The elastic modulus and hardness of feldspar decrease gradually and tend to be stable with the increase of indentation depth
- (3) Under conditions of the same holding time and maximum load, the elastic modulus and hardness of quartz increase with loading rate. The maximum indentation depth and residual indentation depth decrease with the increase of loading rate. The creep displacement increases with loading rate, while the creep compliance decreases with the increase of loading rate. With the increase of holding time, the creep rate decreases and tends to be stable

The research provides reference and basis for studying the microscopic mechanical properties of rock and revealing the microscopic damage mechanism of rock.

Data Availability

All data, models, and code generated or used during the study appear in the submitted article.

Conflicts of Interest

The authors declare no conflict of interest.

Acknowledgments

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