The effect of particle size and hydrate status on dynamic mechanical properties of hydrate-bearing sediments

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Abstract
Dynamic mechanical properties of natural gas hydrate (NGH) reservoirs are extremely important owing to their critical role in drilling safety and reservoir stability during NGH development. However, the comprehensive influence of reservoir skeleton and hydrate status on the dynamic mechanical properties of hydrate-bearing sediments (HBS) has rarely been reported. In this study, three types of sandy sediments with different particle size were simulated. A customized resonant column system was used to synthesize HBS and to test the dynamic mechanical properties of the HBS under the conditions of different confining pressures and shear strains. In addition, the dynamic mechanical responses of the hydrate-free and hydrate-dissociated specimens were comprehensively studied to further demonstrate the dominant role of hydrate status and skeleton-hydrate interactions on the dynamic mechanical properties of HBS. The results show that the shear moduli of the HBS specimens increase exponentially with an increase in confining pressure and decrease in amplitude strain. However, the damping ratios decreases with an increase in confining pressure and decrease in amplitude strain. With an increase in the median particle size (D50) of the simulated sediments, the shear moduli tend to increase, whereas the damping ratios decrease.

In addition, the hydrate occurrence decreases differences in moduli between fine-, medium- and coarse-skeleton specimens, and the enhancement effect of hydrate on the mechanical properties of the specimen is the maximum for the fine sediment specimens. Interestingly, we find that the modulus of the hydrate-dissociated specimen is relatively smaller than that of the hydrate-free specimen, which implies that the structure of the sediments could be rearranged and weakened due to the hydrate formation-dissociation processes.

KEYWORDS
damping ratio, hydrate status, particle size, resonant column, sandy sediments, shear modulus
1 | INTRODUCTION

Natural gas hydrate (NGH) is an ice-like, non-stoichiometric crystalline compound formed by water and gas molecules under low temperature and high pressure, and is also known as “combustible ice”.1-4 NGH is viewed as an alternative energy source because of its high reserves, environmental friendliness and widespread distribution characteristics compared to fossil fuels.5-9 The exploitation of hydrates weakens the mechanical properties of NGH reservoirs, which are likely to cause marine geological problems such as submarine landslides and reservoir subsidence.10-13 Therefore, it is necessary to study the mechanical properties of hydrate-bearing sediments (HBS). Recently, the static mechanical properties of HBS were examined systematically using triaxial tests and direct shear tests, and several scholars have focused on the relationships between the mechanical properties of HBS and confining pressure, hydrate saturation, porosity, strain rate, and temperature, etc.14-21 Nevertheless, natural hydrate reservoirs are usually present in a dynamic environment, such as earthquakes, changes in sea level, and even man-made disturbances (drilling and mining). Therefore, the mechanical properties of HBS, particularly under dynamic loading, are very important for supporting safe drilling and exploitation of NGH.22

Generally, dynamic triaxial test methods, resonant column methods, and wave velocity tests are used to measure the dynamic characteristics of materials.23,24 Clayton et al.25 firstly updated the resonant column into a hydrate synthesis and strength test device, and conducted a series of tests to analyze the effect of hydrate saturation on the shear modulus, bulk modulus, and damping ratio. Priest et al.26 studied the effect of gas hydrate content on P- and S-wave velocities from remote seismic methods. Lee et al.27 used a hydrate wave velocity test device to measure the mechanical properties of HBS under a small strain, providing reference values for calibrating logging and seismic exploration results in hydrate reservoirs. Zhang et al.28 conducted dynamic triaxial shearing tests on tetrahydrofuran (THF) hydrate. Zhu et al.29 studied the dynamic strength characteristics of methane HBS under seismic load, and conducted a series of dynamic triaxial tests on artificial methane hydrate-bearing sediments under various confining pressures between 0.5 and 2.1 MPa. Liu et al.30,31 used a customized hydrate resonant column to test the HBS at different hydrate saturations and identified the relationship between wave velocity, hydrate saturation, and confining pressure.

Based on the above experiments mentioned, the major impacts of effective confining pressure and hydrate saturation on the dynamic characteristics of HBS has been widely studied. However, globally, hydrate reservoirs show wide variations in particle sizes, which can induce different mechanical responses during field hydrate production. Therefore, it is necessary to investigate the dynamic mechanical properties of HBS with different particle sizes. In addition, attention should also be paid to the mechanical properties of HBS before and after hydrate dissociation, as the status of the hydrate reservoir changes substantially during the production processes. This study employed a customized resonant column system to investigate the dynamic mechanical properties of THF hydrate-bearing sediments, hydrate-free sediments, and hydrate-dissociated sediments at small strain ranges. The effects of particle size, confining pressure, and different hydrate status on the shear modulus and damping ratio were studied. These results can provide a reference for the dynamic mechanical characteristic estimations of hydrate reservoirs during production.

2 | EXPERIMENTAL STUDY

2.1 | Experimental apparatus and specimen preparation

A customized resonant column system, developed by the Qingdao Institute of Marine Geology, was used in our study. A schematic of the experimental apparatus is shown in Figure 1. This device was equipped with a water-cooling jacket and a high-pressure chamber to satisfy hydrate formation conditions in porous media. The testing system included five main parts: a cooling system, a pressure loading system, a signal generation system, a drive system, and a data acquisition and processing
system. During the test, the signal generator launched a sinusoidal signal, and the load drive generated motion after receiving the signal, thereby causing the specimens to vibrate. Finally, the signal acquisition and display system perceived the response of the specimen. In the test, shear moduli of the specimens were obtained based on the resonant frequency in the vibration process, and damping ratios of the specimens were measured by free vibration under the resonant frequency. More experimental details can refer to our previous studies.\textsuperscript{30,31}

Figure 2 shows a diagram of the two driving modes. In the torsional mode, the four coils were simultaneously supplied with synchronous currents to make the four magnets rotate horizontally in the same direction, thereby forming a horizontal rotating moment on the top of the specimen to produce torsional shearing motion. In the flexural mode, only two coils were supplied with asynchronous current to cause the corresponding magnets to move horizontally in the same direction, thereby forming a horizontal driving force on the top of the specimen to produce a flexural movement. For the two driving modes, the obtained dynamic mechanics parameters are also different. The shear modulus and damping ratio can be obtained in the torsional mode, and the elastic modulus and damping ratio are obtained in the flexural mode. This study adopted the most conventional torsion mode.

THF hydrate was used as a substitute for NGH, owing to its advantages of operational simplicity, rapid formation, and easy control of hydrate saturation. Several scholars have used THF to synthesize hydrate specimens to characterize their mechanical properties.\textsuperscript{32-36} Coarse, medium, and fine sands were selected as skeleton particles to study the effect of sand particle size on the dynamic characteristics of the HBS. We mixed the three types of sand with a THF solution to synthesize HBS with a hydrate saturation of 30%. Figure 3 shows the morphologies of the three-type sediment particles observed under an optical microscope. The mean particle sizes ($D_{50}$) of the sands were 64.2 $\mu$m (fine sand), 405 $\mu$m (medium sand), and 712 $\mu$m (coarse sand), respectively. Figure 4 shows the particle size distributions of the three types of sand. The mass ratio of THF to water in the aqueous solution was 19:81, and this ratio can theoretically guarantee that the THF solution is completely converted into hydrate.

2.2 Experimental procedures

The tests on each specimen were conducted under different confining pressures (0.5, 1.0, 1.5, 2.0, and 2.5 MPa) and different strains (between $10^{-6}$ and $10^{-4}$). When the hydrate completely dissociated, the test was repeated to analyze the corresponding dynamic mechanical characteristics of the specimen without hydrate. Figure 5 shows a schematic of the experimental procedure. First, keep the reactor space clean and dry, and enclose the rubber film. The mixture of sand and water is then loaded into the mold. Next, the specimen is loaded into the reactor and the temperature of the system is reduced to $-5^\circ$C. After the synthesis of 24 h, the system temperature is increased to 2$^\circ$C, which ensures no ice in the pore. Finally, the resonance tests are performed after the hydrate is synthesized and dissociated, respectively. Hereafter, we refer to a
specimen of the densely packed sand with THF hydrate synthesized using the above-described procedure as a "hydrate-bearing specimen (HBS)." HBS with skeletons of fine sand \( (D_{50} = 64.2 \, \mu m) \), medium sand \( (D_{50} = 405 \, \mu m) \), and coarse sand \( (D_{50} = 712 \, \mu m) \) were denoted as HB1, HB2, and HB3, respectively. The corresponding hydrate-free specimens were denoted as HF1, HF2, and HF3. Similarly, the specimens after hydrate dissociation in the above sand specimens were denoted as HD1, HD2, and HD3, respectively. The compositions of all specimens and the corresponding test conditions are listed in Table 1.

2.3 | Experimental data interpretation

The resonant column test is based on the one-dimensional wave theory, which mainly exhibits dynamic characteristics in a small strain range. Its calculation model assumes that the top of a specimen is free and the lower boundary is fixed. The specimen is vibrated by a drive system, and the resonant frequency of the specimen is measured simultaneously. Subsequently, the dynamic shear modulus of the specimen can be determined by Eq. (1) (Technical Manual for Geotechnical Tests, 2003).

\[
G = \rho \left( \frac{2\pi f}{\beta} \right)^2
\]  

(1)

where \( G \) is the dynamic shear modulus of a specimen, \( \rho \) is the density of a specimen, \( f \) is the resonance frequency in the torsional mode, \( l \) is the height of a specimen, and \( \beta \) is the eigenvalue of the torsional vibration frequency equation. Figure 6 shows the frequency response curve, where the ordinate of the peak corresponds to the resonant frequency.

The damping ratio \( D \) measured in the resonant column test is obtained based on the free decay curve, which is measured by an accelerometer mounted on the resonant column drive disk. Specifically, we applied a sine wave signal to the specimen first, and then stopped the excitation and measured the result of free vibration. Figure 7 shows the typical free vibration decay response curve, and the following equation is used to calculate the damping ratio (Technical Manual for Geotechnical Tests, 2003).

\[
D = \frac{1}{2\pi m} \ln \left( \frac{A_N}{A_{N+m}} \right)
\]  

(2)

where \( D \) is the damping ratio of a specimen, \( A_N \) is the amplitude of \( N \) times, \( A_{N+m} \) is the amplitude of \( (N + m) \) times.

3 | RESULTS AND ANALYZE

3.1 | Dynamic responses of shear modulus and damping ratio with confining pressure

3.1.1 | The shear moduli of specimens

Figure 8(A)–(C) displays a logarithmic relationship between the shear modulus and confining pressure for different sediment particle sizes. It is clear that the shear modulus of all specimens exponentially increase with an increase in confining pressures, because the confining pressure can increase the friction between the particles.
and lock the particles to strengthen the shear moduli of specimens. In addition, the increase in the shear modulus under low confining pressures is more rapid than that under high confining pressures. This is mainly because microstructures of the specimens gradually become denser under higher confining pressures, resulting relatively less reduction in pore sizes.16,37-40

Comparing differences in shear moduli between specimens with different particle sizes, we find that the shear modulus of HB3 is the highest among the three hydrate-bearing specimens, whereas HB1 has the lowest shear modulus. However, the shear strength of the hydrate-bearing specimen is significantly improved as compared to that of the hydrate-free and hydrate-dissociated specimens, as the formation of hydrate reduces the porosity and increases the overall stiffness of the specimen. After hydrate dissociation, the shear modulus shows a slightly decreasing trend compared to that of the hydrate-free specimen, which may be related to the damage of the sand skeleton after hydrate dissociation.39,40 In addition, the shear moduli of HB1, HB2, and HB3 with identical hydrate saturation and under the same confining pressure are similar. However, the shear moduli of the other specimens without hydrate (HF1, HF2 and HF3) or after hydrate dissociation (HD1, HD2 and HD3) show a significant difference. This phenomenon demonstrates that both skeleton particles and hydrate influence the dynamic mechanical properties of HBS, and the hydrate is the dominant factor and decrease the influence of skeleton particles on the dynamic mechanical properties of HBS.

To further understand the influence of the hydrate status on the shear modulus of the specimens, the results presented in Figure 8(A)–(C) were redrawn with categories of fine Figure 8(D), medium Figure 8(E), and coarse Figure 8(F) sediment specimens. It is observed that the shear moduli of hydrate-bearing specimens are

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**TABLE 1** The composition of each group specimen at the corresponding test conditions

| No. | Specimen states               | Porosity n (%) | Particle size | Confining pressure (MPa) | Strain Y (σ = 2.5 MPa) |
|-----|-------------------------------|----------------|---------------|--------------------------|------------------------|
| HF1 | Hydrate-free specimen         | 0.41           | Fine sand     | 0.5–2.5                  | 10^{-6}–10^{-4}         |
| HF2 |                               | 0.41           | Medium sand   | 0.5–2.5                  | 10^{-6}–10^{-4}         |
| HF3 |                               | 0.41           | Coarse sand   | 0.5–2.5                  | 10^{-6}–10^{-4}         |
| HB1 | Hydrate-bearing specimen (S_h = 30%) | 0.41         | Fine sand     | 0.5–2.5                  | 10^{-6}–10^{-4}         |
| HB2 |                               | 0.41           | Medium sand   | 0.5–2.5                  | 10^{-6}–10^{-4}         |
| HB3 |                               | 0.41           | Coarse sand   | 0.5–2.5                  | 10^{-6}–10^{-4}         |
| HD1 | Hydrate-dissociation specimen | 0.41           | Fine sand     | 0.5–2.5                  | 10^{-6}–10^{-4}         |
| HD2 |                               | 0.41           | Medium sand   | 0.5–2.5                  | 10^{-6}–10^{-4}         |
| HD3 |                               | 0.41           | Coarse sand   | 0.5–2.5                  | 10^{-6}–10^{-4}         |
the highest, and the shear moduli of all hydrate-free specimens are higher than those of hydrate-dissociated specimens under the same confining pressures. The presence of hydrates contributes to the mechanical properties of the specimens, and this enhancement effect is the maximum in the fine grain specimens.

3.1.2 The damping ratio of specimens

The relationships between the damping ratios and confining pressures are shown in Figure 9(A)–(C). It can be seen that the damping ratios of the HBS decrease dramatically with an increase in the confining pressures.
This phenomenon occurs mainly because the energy loss of wave propagation reduces when the confining pressure increases and causes the specimen particles move close. Similar relationships appear in the hydrate-free and hydrate-dissociated specimens. In addition, regardless of pore states in the hydrate-free, hydrate-bearing, and hydrate-dissociated specimens, the damping ratios of the specimens show an opposite trend to that of the $D_{50}$. Previous studies have shown that the mechanical properties of HBS are related with the specific surface area of the particles. Normally, a larger specific surface area contributes to relatively poor mechanical properties of HBS.41
The specific surface area of the fine particles is the larger, and the contact areas between the hydrate and sand particles in the fine specimen are greater than those of the coarse specimen. Thus, the wave energy is more easily consumed in the process of propagation.

Similarly, the damping ratios of the specimens presented in Figure 9(D)–(C) were redrawn with categories of fine Figure 9(D), medium Figure 9(E), and coarse Figure 9(F) sediment specimens. It is observed that the hydrate-dissociated specimens show the highest damping ratio, while the hydrate-free specimens show the lowest one. This may be mainly caused by the different amount and distribution of pore water before and after hydrate dissociation. In other words, the existence of a high amount of pore water increases the energy consumption during shear wave transmission. According to Phol et al., water is present on the surface of hydrates above the freezing point, and the existence of liquid water between hydrate particles is likely to cause high attenuation. Similarly, the formation of hydrates and the particle sizes both affect the specimen damping ratios according to Figure 9.

### 3.2 The stress sensitivity of shear modulus

According to the results shown in Figure 8, the following equation is used to fit the data in Figure 8.

\[ G = a \cdot \sigma^b \] (3)

where \( \sigma \) is the confining pressure, \( a \) is the shear modulus under unit stress, and \( b \), the stress sensitivity index, represents the sensitivity of shear modulus to confining pressure, which is usually equal to 0.5 for granular materials under small shear strain conditions.

### 3.3 Dynamic response of shear modulus and damping ratio with shear strain

#### 3.3.1 The shear moduli of specimens

As shown in Figure 11(A)–(C), the relationship between the shear modulus and shear strain is affected by the sediment particle size. We find that the shear moduli of the HBS decrease with an increase in shear strain. This behavior may be caused by micro-cracks or damages of the specimen gradually develop as the shear strain increases, resulting in a corresponding decrease in the shear modulus.
modulus. Similar to the case of the modulus shown in section 3.1.1, the specimens with a large sand particle have a large shear modulus under identical strain. In addition, the modulus variation is the smallest when hydrate is present in the specimen. The overall trend of relationship between the modulus and shear strain is broadly consistent with that between the modulus and confining pressure as shown in Figure 8. It is clear that the shear moduli of HBS are the largest, and the hydrate dissociation specimens are the smallest regardless of the type of sand adopted.

3.3.2 The damping ratios of specimens

Figure 12(A)–(C) displays the relationship between the damping ratio and shear strain under different sediment particle sizes. (D)–(F) modulus data of hydrate-free, hydrate-bearing and hydrate-dissociated specimens.
sizes. The results indicate that the damping ratios of the HBS increase with an increase in the shear strains. In addition, the damping ratios of all HBS specimens show an opposite trend to that of $D_{50}$ in Figure 4, implying that the sediment particle size has a significant influence on the damping ratio of the HBS. In addition, Figure 12(D)–(F) shows the relationship between the damping ratio and shear strain of hydrate-free, hydrate-bearing and hydrate-dissociated specimens. The results show that the damping ratio of the HBS increases with an increase in shear strain. One possible reason for this is similar with that for the shear modulus, i.e., the micro-cracks in the specimen gradually increase with the increase in shear strain, which causes the specimens to gradually become loose, increasing the shear wave energy attenuation rate.
3.4 The normalized shear modulus $G/G_{\text{max}}$

The relationship between the normalized shear modulus and shear strain is called the shear modulus degradation curve, which reflects the degradation behavior of shear modulus with an increase in strain. To clarify the variation in shear modulus under different shear strains, the Hardin-Drnevich model is fitted based on Eq. 3.46:

$$\frac{G}{G_{\text{max}}} = \frac{1}{1 + \frac{\gamma}{\gamma_{\text{ref}}}}$$  \hspace{1cm} (4)

where $G$ is the shear modulus, $G_{\text{max}}$ is the maximum value of the shear modulus, $\gamma$ is the shear strain, and $\gamma_{\text{ref}}$ is the reference value of shear strain.

In general, the shear modulus degradation curve is divided into four stages: linear, degradation, critical, and residual stages. Owing to the range limitation of strains available in our resonant column tests, the shear modulus degradation curve here includes the linear and degradation stages only. Comparing the degradation curves of specimens before and after hydrate dissociation, as shown in Figure 13(A) and (B) respectively, it can be observed that the particle size has a random effect on the shear modulus degradation curve of the specimens. Furthermore, the linear stage of the degradation curve of the hydrate-dissociated specimens lasts longer than that of the hydrate-bearing specimens shown in Figure 13(C) and (D). One possible reason for this is that the normalized shear modulus of dense specimen is small under the same conditions. The hydrate particles fill the pores among the sand particles during the THF hydrate formation, which increases the shear modulus of HBS and simultaneously reduces the effective porosity of the sediment. Therefore, the linear stage of the hydrate-bearing specimens is relatively short.

**FIGURE 13** The shear modulus degradation curves of all specimens. (A) Degradation curves of HB1-HB3. (B) Degradation curves of HD1-HD3. (C) Degradation curves of HB1 and HD1. (D) Degradation curves of HB2 and HD2
4 | DISCUSSION

4.1 | Effect of the particle size

The shear modulus increases while the damping ratio decreases with the increase in sediment particle size according to Figure 8-9, which may be attributed to the micro-crack decrease with increasing particle size. In order to elucidate this reason clearly, we further built particle flow code (PFC) models for the three type specimens. Figure 14(A)-(C) shows the structures of the specimens with different particle sizes, where the red contact lines are represented as cracks. It can be seen that as the particle size decreases, the number of such cracks between particles increases. Previous studies also demonstrate that the smaller the particle size is, the weaker the macro-mechanical characteristics is, the strength of coarse sediments is greater than that of fine ones under the same specimen volume condition.24,38,48-50 Figure 14(D) shows that $D_{50}$ and shear modulus have a linear upward trend within a certain range. However, $D_{50}$ and damping ratio show an exponential decrease trend in Figure 14(E).

For the hydrate-free and hydrate-dissociated specimens, the friction and occlusion forces of the sediment particles played a dominant role in shear strength. However, for the hydrate-bearing specimen, the cementation hydrate can significantly improve the shear strength.

4.2 | Effect of hydrate status

As the results showed in Figure 8, the shear moduli of the hydrate-bearing specimens is the highest compared to those of the hydrate-dissociated and hydrate-free specimens, which may be attributed to cementation forces between the sand and hydrate particles. We also find the shear moduli of the hydrate-dissociated specimens are lower than those of the hydrate-free specimens. The possible reasons for this are as follows: (1) the dissociated pore water cannot be discharged immediately, which leads to an increase in pore pressure and a decrease in effective stress, thus reducing the shear modulus; (2) pore water also acts as a lubricant between particles, reducing frictions between particles; and (3) the originally densely-packed sediment particles become loose after hydrate dissociation. However, for the damping ratio, the hydrate-dissociated specimens show the highest values, and the hydrate-free specimen show the lowest ones. These behaviors may be related to the different pore water contents of the specimens under different hydrate status conditions. And the viscous motion of the pore water accelerates energy consumption of the shear wave passing through the particles51, thus increasing the damping ratio. For the hydrate-dissociated specimens, they contain the highest amount of pore water, and thus, the damping ratios are the highest. The damping ratio

![Figure 14](image-url)
values of the hydrate-bearing specimens are between those of the hydrate-dissociated and hydrate-free specimens, which can be explained by the fact that the THF solution can not be completely converted into hydrate, and the water in the residual solution will remain in the specimens. Figure 15 shows the whole evolution of the dynamic mechanical parameters measured in this study. In general, both the particle size and hydrate status affect the dynamic mechanical parameters of the specimens. The shear modulus exhibits an increasing trend with the increase in hydrate occurrence and particle size. However, the influencing factors of the damping ratio include pore water and particle size distribution, wherein pore water distribution could be a dominant factor.

5 | CONCLUSION

In this study, a series of resonant column tests were conducted to investigate the comprehensive effect of particle size and hydrate status on the dynamic mechanical properties of hydrate-bearing sediments. Based on the experimental results, the main findings of this study are summarized below.

1. The shear modulus of HBS increases with an increase in confining pressure. However, the damping ratio shows an opposite trend. In addition, the damping ratio of HBS increases with an increase in shear strain, and the corresponding shearing modulus decreases at the same time.

2. The shear modulus of the hydrate-dissociated specimen is relatively weaker than that of the hydrate-free specimen, which implies that the structure of the sediments could be weakened by the hydrate dissociation processes.

3. The particle size significantly influences the dynamic mechanical properties of HBS under a smaller strain. The larger the particle size is, the greater the mechanical properties of HBS are. Additionally, the presence of hydrate shows more pronounced in improving shear modulus of the fine-specimens compared to that of medium- and coarse-sediments. Thus, the hydrate can minimize the influence of particle size on the dynamic mechanical properties of HBS. However, the particle size becomes the dominant factor again, when the hydrate is dissociated in specimens.

4. The high pore water content is the main reason for high damping ratio of the hydrate-dissociated specimen. The more the pore water amount is, the greater the energy consumed by the wave propagation process is, which results in a large damping ratio.

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