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PHYSICAL PROPERTIES OF REPRESSURIZED SAMPLES RECOVERED DURING THE 2006 NATIONAL GAS HYDRATE PROGRAM EXPEDITION OFFSHORE INDIA

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ABSTRACT

As part of an international cooperative research program, the U.S. Geological Survey (USGS) and researchers from the National Gas Hydrate Program (NGHP) of India are studying the physical properties of sediment recovered during the NGHP-01 cruise conducted offshore India during 2006. Here we report on index property, acoustic velocity, and triaxial shear test results for samples recovered from the Krishna-Godavari Basin. In addition, we discuss the effects of sample storage temperature, handling, and change in structure of fine-grained sediment.

Although complex, sub-vertical planar gas-hydrate structures were observed in the silty clay to clayey silt samples prior to entering the Gas Hydrate And Sediment Test Laboratory Instrument (GHASTLI), the samples yielded little gas post test. This suggests most, if not all, gas hydrate dissociated during sample transfer. Mechanical properties of hydrate-bearing marine sediment are best measured by avoiding sample depressurization. By contrast, mechanical properties of hydrate-free sediments, that are shipped and stored at atmospheric pressure can be approximated by consolidating core material to the original in situ effective stress.

Keywords: acoustic velocity, disturbance, friction angle, pressure cores, shear strength

NOMENCLATURE

A - change in pore pressure/change in deviator stress [kPa/kPa]
c/p - shear strength/consolidation stress [kPa/kPa]
Ms - mass of solids [g]
Msw - mass of seawater [g]
Mt - total mass of sediment sample [g]
p' - normal effective stress acting on a plane inclined at 45° from the horizontal, $(\sigma'_1 + \sigma'_3)/2$ [kPa]
q - shear stress acting on a plane inclined at 45° from the horizontal, $(\sigma_1 - \sigma_3)/2$ [kPa]
Vp - acoustic P-wave velocity [km/s]
φ'max - maximum friction angle in terms of effective stresses, passes through the origin [degrees]

INTRODUCTION

As part of an extensive international program to study gas hydrates offshore India during 2006 [1], pressure cores were recovered and preserved at near in situ conditions. Three whole-round, chilled, pressurized sub-samples from the Krishna-Godavari Basin, located along the eastern

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continental margin of India, were shipped to the USGS Gas Hydrate And Sediment Test Laboratory Instrument facility. The sediment was recovered from Hole NGHP-01-21A (15° 51.8531'N; 81° 50.0827'E; water depth: ~1049 m) which was cored between 48 and 91.5 mbsf. The samples were obtained at a subbottom depth of 58 to 59 mbsf in pressure core 2Y. The 1-m long core had an 84% sediment recovery [1]. Site NGHP-01-21 was the last region occupied during the NGHP-01 cruise, was located near previously drilled sites 10, 12, and 13, and showed strong evidence of gas hydrate accumulations. Core recovery at Site NGHP-01-21 was better than in previously drilled nearby holes at the same depth interval [1].

The regional structural setting is interpreted to be a highly disturbed or faulted sedimentary sequence overlying high-amplitude deep gas deposits [1]. Individual seismic reflectors are lost below a few hundred m subbottom. The recovered sedimentary sequence is interpreted to be a single lithostratigraphic unit. The recovered sediments were typically black-colored clays containing terrigenous organic matter, authigenic carbonate and thin intervals of pyrite, foraminifera, and plant debris [1]. Gas hydrates in the Krishna-Godavari Basin often occupied a fractured reservoir network or were present in the pore space of silty clays or sand and silt beds [1].

A triaxial-test facility to measure physical properties of never-depressurized pressure-core samples does not exist. Therefore, we measured physical properties of the samples after they were transferred into GHASTLI at atmospheric pressure, and then repressurized to approximately in situ conditions. Accurate physical property measurements of natural gas-hydrate-bearing sediment are needed for models that predict behavior during exploration, drilling, and production projects. However, this is not easy to achieve because natural gas hydrate dissociates when removed from pressure and temperature conditions in which it is stable, and effective stresses within the sediment change in response to coring and storage conditions.

Natural-hydrate-bearing coarse-grained sediment, that was stabilized by freezing during the recovery process, was previously tested in GHASTLI [2]. Freezing of the pore water reduced sediment disturbance during transport of the samples and reduced gas hydrate dissociation during transfer into GHASTLI. We contemplated freezing natural hydrate-bearing NGHP-01 samples to facilitate their transfer into GHASTLI, also. However, the effect of freezing on fine-grained sedimentary structure was unknown. Therefore, we performed a preliminary study to determine if freezing could be used with fine-grained sediment. Computed axial tomography (CAT) imaging documented the effect of different freezing rates on marine sediment structure.

Triaxial tests are used for measuring acoustic velocity, determining shear strength and moduli, and observing behavior under in situ effective stress conditions that are currently not routinely available by well logging or borehole testing. Properties of "undisturbed" samples containing natural gas hydrate are especially important because of the difficulty in forming complex gas hydrate structures within fine-grained sediment in a laboratory. Unfortunately, all actual samples are disturbed to some degree and an understanding of the degree of disturbance is critical to data interpretation. These results complement pressure-core studies conducted on the same sediment and physical property measurements made at sea on sediment from nearby holes [1].

FIELD AND LABORATORY EQUIPMENT

The whole-round sediment samples tested in GHASTLI were recovered at close to in situ pressure during the NGHP-01 cruise off India using the Fugro Pressure Corer that utilizes a water hammer technique to drive a sample barrel ahead of the drill bit into sediment with shear strength less than or approximately 500 kPa [1]. The samples were transferred under pressure into storage chambers at sea and acoustic P-wave velocity, gamma ray attenuation, and x-ray image data were collected using the Geotek Pressure Multisensor Core Logger (MSCL-P) [1]. The pressurized samples were cut, post cruise, into shorter lengths in Singapore, transferred under pressure into smaller pressure vessels that were sealed with ball-valve assemblies (Figure 1), then transported to Woods Hole via refrigerated air freight [3], and were stored at +4°C until testing.

The primary system used during testing is the Gas Hydrate And Sediment Test Laboratory Instrument (GHASTLI) [4], which simulates in situ pressure and temperature conditions on cylindrical sediment samples (Figure 2). A bath circulator is used to control the temperature of the sample chamber and of a heat exchanger located immediately above the top specimen end cap. Four

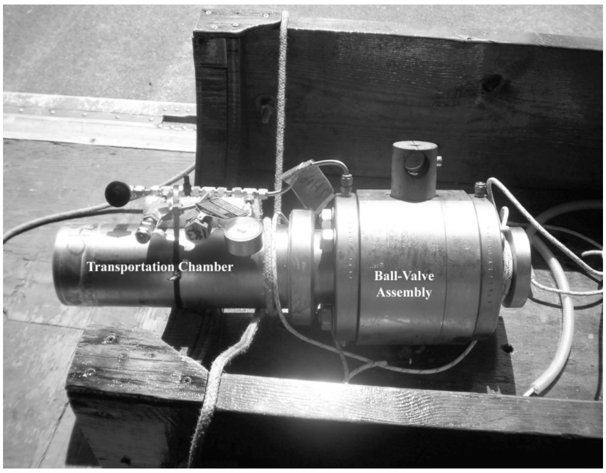


Figure 1. Pressure transportation vessel (smaller-diameter section at left) containing a NGHP-01 sample, sealed with a ball-valve assembly (larger-diameter section at right).

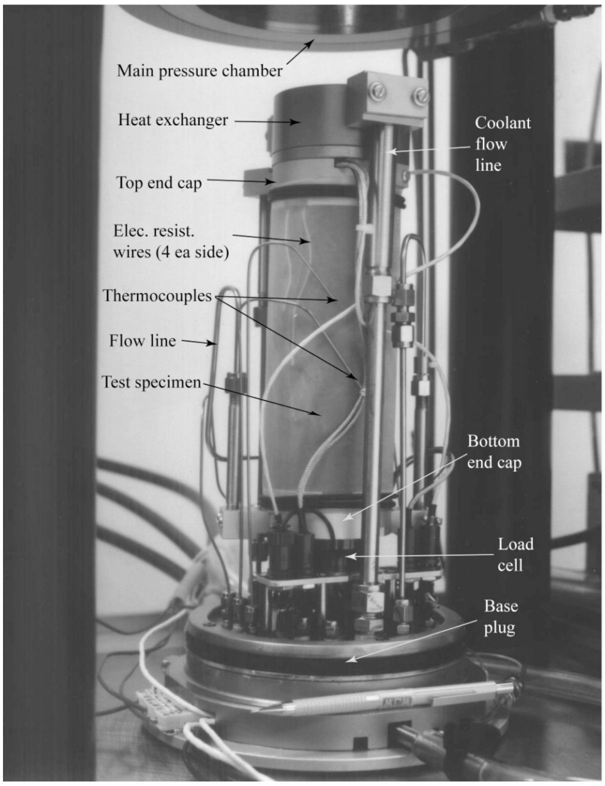


Figure 2. Close-up view of a test specimen about to be raised into the main pressure vessel (visible at the top of the photograph). The test specimen (gray cylinder) is located in the central part of the photo and rests on an interchangeable internal load cell. A heat exchanger that imparts a unidirectional cooling front downward through the specimen rests atop the upper end cap, fed through the large diameter, vertical tubes at the front and rear of the specimen [4].

thermocouples and four thermistors are placed against the outside perimeter of the specimen or end caps at different heights to measure temperature variations along the sample surface.

Three separately controllable 500-ml-capacity syringe pumps are used to maintain the confining pressure surrounding the specimen and internal specimen pressures. A back-pressure system contains a collector capable of separating and measuring water and gas volumes which are pushed out of the specimen by gas hydrate dissociation at test pressures [4]. A separate, fourth, syringe pump controls the movement of the load ram during the shear phase of the test. The ram position is used to determine the specimen height. Load, pressure, temperature, and acoustic measurements from within the different subsystems and in close proximity to the test specimen are logged and displayed by a computer employing custom-designed Labview software.

EXPERIMENTAL METHODS

Sample handling methodology

Sediment samples containing natural gas hydrate cannot be transferred directly under pressure into GHASTLI because it does not have a mechanism that mates with the transportation ball-valve assembly. Such a mechanism is more than one m long [1] and cannot fit inside the main test chamber. Hence, pressure-core samples must be depressurized. However, depressurizing natural gas hydrate initiates dissociation which can significantly change the overall sample physical properties [5]. Reducing the sample temperature helps stabilize hydrate by bringing it closer to the stability field, thereby mitigating some of the detrimental effects of depressurization.

We investigated methods of freezing sediment that could be used to reduce the dissociation of natural gas hydrate within sediment during the transfer from ball-valve-sealed pressure vessels into GHASTLI. But first we needed to determine if freezing had an effect on structure of fine-grained sediment. We froze three marine sediment samples obtained from off the east coast of the United States. Two samples were frozen at different rates in a walk-in freezer and an additional sample was frozen by immersing it in liquid nitrogen (LN_2). CAT images were made of the three samples before and after freezing. We did not freeze any samples recovered from offshore India.

A secondary goal of the program was to determine the effect of side filter drains on the consolidation characteristics of sediment within GHASTLI. Side drains can be used to decrease the time required for consolidation and also to equilibrate pore pressure during shear. However, installing side drains is time consuming and complicates the handling process during the transfer of samples from pressure vessels into GHASTLI.

The approach we used to transfer a NGHP-01 sample from a pressurized transportation chamber into GHASTLI began by venting the flammable methane gas in the ball-valve-sealed vessel outdoors, next to the building that housed the test facility. Based on what we learned from the study of freezing effects on fine-grained sediment, the test sample was kept refrigerated and was not frozen. However, to help minimize gas hydrate dissociation, the depressurized transportation pressure chamber (without the ball-valve assembly) and sample were quickly brought into the laboratory and placed in a top-loading freezer. The sample, encased in a clear plastic liner, was removed from the pressure vessel. A longitudinal slice was made only in the liner with a rotary-bit-cutting tool. The sediment was not cut. The liner was expanded and removed from the sample. The ends of the sample were trimmed with a miter box and wire saw or a "chop" saw. Side-filter drains were applied, followed by a flexible membrane that was wired to a top and bottom end cap. The sample was quickly placed into GHASTLI where the chamber surrounding the test sample was flooded with chilled confining fluid. Pressurization of confining and sample-pore fluids completed the process.

Methods used for NGHP-01 samples

Index properties

Specimens used for index property calculations were dried at a temperature of 105°C for at least 24 hours to determine the amount of fresh water and solids present. The volume of dried solids was determined with an automatic gas pycnometer using helium as the purge and expansion gas [6]. The grain density of the pycnometer specimen was calculated using the mass of solids as determined immediately prior to insertion of the sample into the pycnometer.

As appropriate, physical-property calculations, assuming pore water salinity of 35 ppt, were corrected for the presence of residual salt left on the solid particles after oven drying. In the natural environment, salt and other particles are dissolved in the pore fluid and behave as part of the aqueous phase. The calculations remove the salt precipitate from the solids and add it back to the fluid phase. Previously published equations are used to calculate grain density, bulk density, porosity, and water content of the sediment [7].

Grain-size analyses

Less than 1 g of wet sediment (grains less than 2 mm diameter) was sonicated in a slurry and flushed through a model 13320 Beckman Coulter Laser Diffraction Particle Size Analyzer to produce a grain-size distribution curve from which statistical parameters were obtained.

Acoustic velocity

P-wave velocity was measured by pulse transmission through the cylindrical sample using 1.1 MHz (natural frequency) wafer-shaped crystals that are located on the back side (away from the specimen) of each end cap. A pulse as high as 100-volts was sent to the transmitting transducer, the received signal was amplified, digitized, displayed on a digital oscilloscope, and recorded by a computer. Acoustic P-wave velocity (Vp) was calculated from the specimen length and measured acoustic travel time through the specimen.

Shear strength

During the shear strength phase of the test, specimen loading was produced by a ram contacting the heat exchanger which pushed on the sample. Samples were sheared at a constant rate (measured using a linear displacement transducer) that was slow enough to ensure equalization of pore pressure throughout the test specimen [8]. Load, confining pressure, pore pressures at the top and bottom end caps, and sample deformation (to a maximum of 15 to 20 percent axial strain) were measured and recorded.

RESULTS AND DISCUSSION

Sample handling methodology

The structure of fine-grained sediment at refrigerated (+4°C), typical freezer (-22°C), and liquid nitrogen (-196°C) temperatures are strikingly different. Refrigerated sediment shows little cracking or disturbance, and appears uniform in CAT-scan images (Figure 3). Compare this internal structure with three frozen samples (Figure 4) where the degree of disturbance and cracking is related to the time required for freezing. The slower freezing rate allows pore water to migrate towards freezing fronts thereby developing more extensive ice-lens patterns.

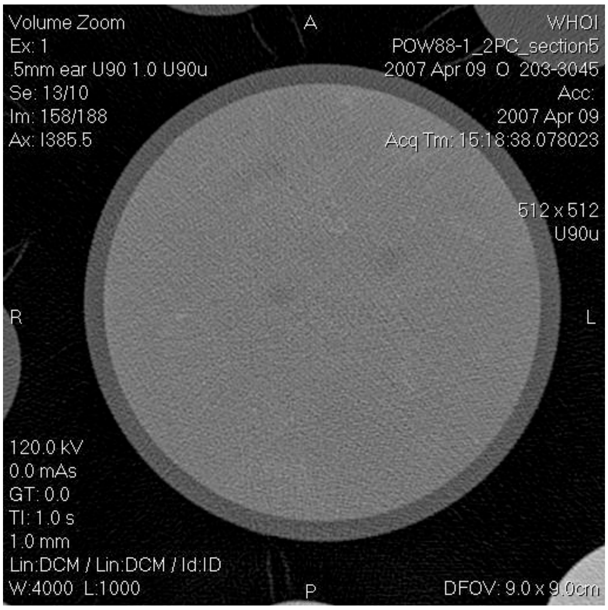


Figure 3. CAT-scan image of a refrigerated (+4°C) POW88-1 (Gulf of Maine) core section. Notice the uniform sediment structure prior to freezing.

Rapid freezing to -196°C in LN_2 does not appear to disrupt fine-grained sediment structure as severely as slow freezing to -22°C, however, radial fractures were noticed on CAT-scan images (center image, Figure 4). A previous study [9] cast doubt on the use of LN_2 for preserving gas hydrate in samples that will be removed from the LN_2 as part of the test procedure.

Upon immersion in water, significant quantities of methane gas were released from trimmed sediments stored at subfreezing temperature and atmospheric pressure for five hours. This may attest to the benefit of keeping gas hydrate frozen and/or the ability of ice converted from hydrate to trap gas molecules [10]. However, results from the sample freezing study indicate that fine-grained sediment, unlike coarse-grained samples from the Mackenzie Delta, NWT [2], should not be frozen because of the extensive disturbance caused by ice-lens formation.

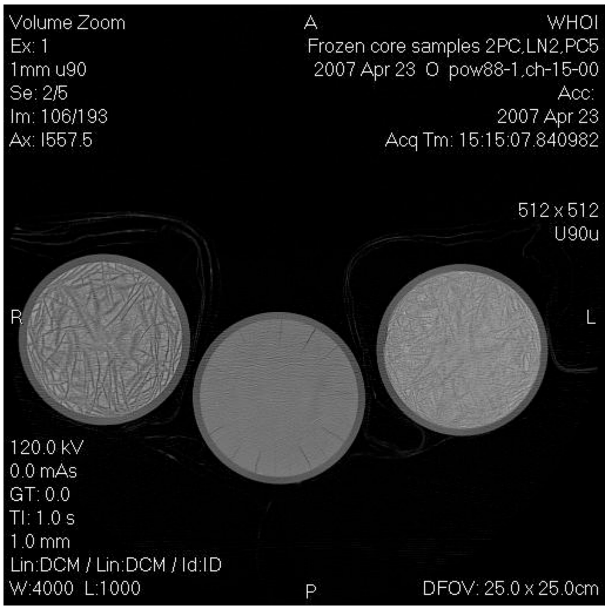


Figure 4. CAT-scan image of three frozen samples: POW88-1 sample from the Gulf of Maine stored within a pressure vessel and slowly frozen at -22°C within a walk-in freezer (left), POW88-1 sample very quickly frozen in LN_2 (middle), and CH-15-00 sample from the Blake Ridge quickly frozen at -22°C within a walk-in freezer (right). Compare the extensive crack pattern in the slowly frozen POW88-1 sample (left) with the radial cracks in the quickly frozen POW88-1 sample frozen in LN_2 (center), but otherwise without major noticeable disturbance.

Refrigerated cores maintained at elevated pressure have the potential to preserve existing natural gas hydrate (Figure 5). However, depressurization of those cores typically imparts significant detrimental effects on fine-grained sediment structure [11]. In addition, depressurization to atmospheric pressure, combined with even short times when temperatures are above freezing have the potential to dissociate most if not all gas hydrate. Due to increased strength and stiffness [12], coarse-grained, hydrate-cemented or frozen sediments are not as significantly disturbed by depressurization as are fine-grained sediments.

Figure 5. NGHP-01 sediment and gas-hydrate veins preserved within a refrigerated, methane-pressurized chamber.

Index, acoustic, and strength properties of NGHP-01 samples

Bulk index properties, including water content, are important because they often correlate to sediment behavior [13, 14] and reflect the degree of compaction and stress history at various subbottom depths. Initial water contents (based on sediment mass) are uniform and vary from a high of 61% for GH117 to 55% for GH115 (Table 1). These values are consistent with the 58 mbsf burial depth of the samples.

Porosity values (62% to 59%) are also uniform for these three immediately adjacent test samples. Because porosity is a measure of the relative volume of the pore space in a sample and is independent of any particular pore-filling material, unlike water content, porosity measurements provide a means for comparing sample attributes. Previous studies have shown that for similar sediment types and test conditions, higher porosity specimens generate more positive pore pressure during shear, are weaker, and can have lower acoustic velocities than a sample with lower porosity [15, 16].

The uniformity of the index properties, which are determined on disturbed sub-samples, suggest that velocity and strength properties should also vary little between test samples. However, the three samples, were disturbed differently during the transfer to GHASTLI. Two samples (GH115 and GH117) had clean breaks through the entire sediment cross-section caused by gas expansion. The other (GH116), remained intact, but swelled and required significant manipulation to complete the transfer.

P-wave velocities varied from 1.56 to 1.90 km/s (Table 1), and increased with effective stress (0 to 400 kPa) and decreasing porosity in agreement with standard trends [17]. However, disturbance lowered the measured Vp values for GH116.

Shear strength related plots are shown in Figures 6 to 11. The adjacent ball-valve-sealed samples exhibit similar contractive behavior during shear as evidenced by positive-pore-pressure build up (Table 1; Figures 6, 8, 10) and stress paths that curve to the left (Figures 6, 8, 10). The positive pore-pressure response is in agreement with other fine-grained samples tested in GHASTLI [18]. After consolidation to 400 kPa, samples GH115 and GH116 also had similar stress – strain properties (Figures 6 and 8), strength/consolidation ratio (0.43) (Table 1), and peak friction angles (30 to 31 degrees, assuming no cohesion intercept) (Table 1; Figures 7 and 9), despite having different degrees of initial disturbance. Maximum shear strength of about 175 kPa developed between 6 and 10 percent strain. The measured strength ratio is higher than expected for a typical fine-grained sediment [19]. Evidently, consolidation to identical stress states mitigates differences in coring and handling disturbance. Similarities in the mechanical properties of GH115 and GH116 suggests that effective stress exerts a primary control on behavior despite significant differences in initial sample integrity.

GH117 had a slickensided shear plane that may have contributed to a lower shear strength (119 kPa), strength/consolidation ratio (0.28), and peak friction angle (25 degrees) (Table 1; Figures 10 and 11). Cracks and higher initial water content

Test Number	GH115	GH115	GH115	GH116	GH116	GH116	GH117	GH117	GH117
	Initial	Pre-shear	Post shear	Initial	Pre-shear	Post shear	Initial	Pre-shear	Post shear
General information									
Project	NGHP-01	NGHP-01	NGHP-01	NGHP-01	NGHP-01	NGHP-01	NGHP-01	NGHP-01	NGHP-01
Core section interval (m)	0.44-0.64	0.44-0.64	0.44-0.64	0.04-0.24	0.04-0.24	0.04-0.24	0.24-0.44	0.24-0.44	0.24-0.44
Subbottom depth interval (mbsf)	58.44-58.64	58.44-58.64	58.44-58.64	58.04-58.24	58.04-58.24	58.04-58.24	58.24-58.44	58.24-58.44	58.24-58.44
Index properties									
Water content (Msw/Ms) (%)	54.8	37.8	39.5	58.26	47.34	46.5	60.8	50.5	43.7
Water content (Msw/Mt) (%)	35.4	27.4	28.3	36.81	32.13	31.8	37.8	33.5	30.4
Grain density (g/cm^3)	2.7	2.7	2.7	2.72	2.72	2.72	2.72	2.72	2.72
Bulk density (calc.) (g/cm^3)	1.71	1.76	1.77	1.69	1.77	1.78	1.67	1.75	1.81
Porosity (calc.) (%)	59.1	52.6	53.0	60.7	55.5	55.3	61.7	57.3	53.7
Grain size									
Mean (phi)	5.92			8.31			8.74		
Median (phi)	6.08			9.60			9.98		
Mean/Median ratio	0.97			0.87			0.87		
Standard Deviation	3.35			3.53			3.87		
Skewness	0.00			-0.25			-0.18		
Kurtosis	-0.73			-0.83			-0.84		
Velocities (GHASTLI)									
Vp (km/s) [consolidation stress (kPa)]	1.781 [0]			1.56 [0]					
Vp (km/s) [consolidation stress (kPa)]	1.818 [100]			1.575 [100]			1.629 [100]		
Vp (km/s) [consolidation stress (kPa)]	1.856 [200]			1.599 [200]			1.663 [200]		
Vp (km/s) [consolidation stress (kPa)]	1.903 [400]			1.634 [400]			1.700 [400]		
Shear properties (GHASTLI)									
Consolidation stress (kPa)			399.04			408.45			416.81
Strain rate (%/hour)			0.129			0.139			0.142
A (failure)			0.65			0.604			1.08
q (failure) (kPa)			172.76			176.75			118.51
p' (failure) (kPa)			347.37			371.81			279.98
c/p ratio			0.433			0.43			0.284
Axial strain (failure) (%)			9.98			5.67			13.80
ϕ'max, friction angle (deg.)			30.16			30.95			25.2
Secant modulus at 1% strain (MPa)			13.35			13.1			6.01

Table 1. Index properties, acoustic velocities, and shear strength properties of sediment recovered from National Gas Hydrate Project Expedition 01 (NGHP-01), core 21A, section 2Y and tested in GHASTLI.

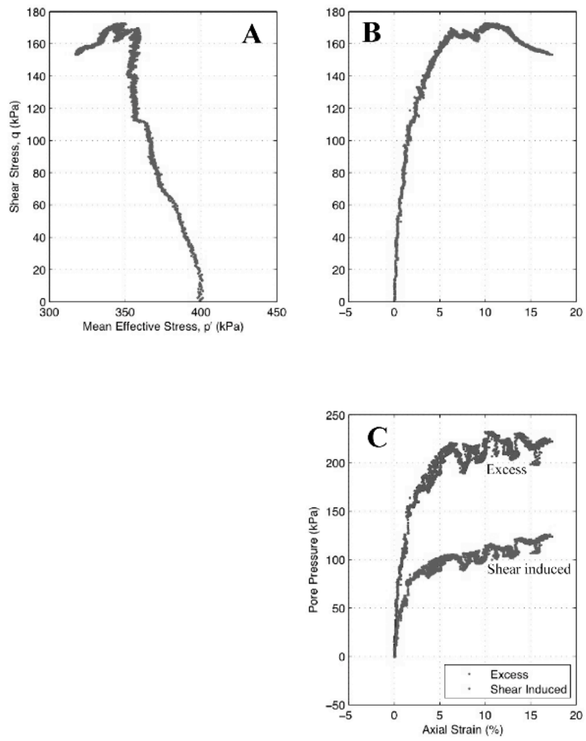


Figure 6. Shear strength results for NGHP-01 test sample GH115. Individual plots are (A) shear stress (q) vs. effective normal stress (p') on a plane inclined at 45° from the horizontal. Each data point represents the top of a Mohr's circle and together they define a "stress path"; (B) shear stress (q) vs. axial strain, and (C) change in relative pore pressures vs. axial strain. At the start of shear, plot A begins in the lower right corner on the horizontal axis and moves to the left, whereas plots B and C start in the lower left corner of the horizontal axis at 0% strain and move to the right.

(61%) may have caused the sample's relative weakness. The slickensided shear plane (oriented at 61 degrees from the horizontal) differs markedly from a nearby thin layer of coarse-grained (coarse silt/very fine sand) sediment, also in GH117, inclined at 32 degrees from the horizontal. The coarser-grained layer may have enhanced the formation of previously observed hydrate veins.

Seating variances between the sample end caps and load ram may be responsible for changes in slope noticed on some plots (e.g., Figures 10 and 11) and scatter in secant modulus values below 1% strain (Figures 7, 9, 11).

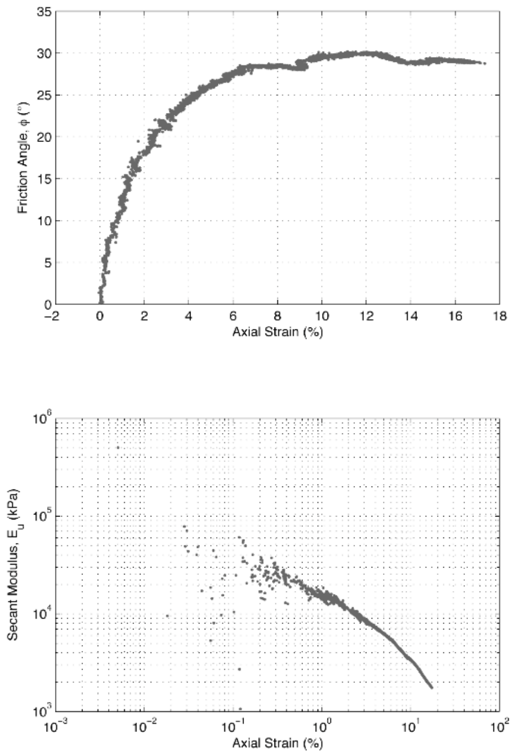


Figure 7. Effective friction angle and secant modulus vs. strain for NGHP-01 sample GH115.

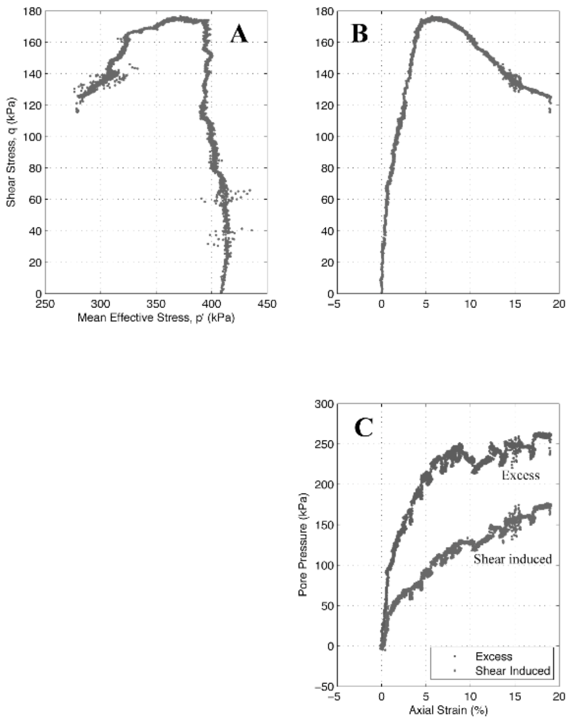


Figure 8. Shear strength results for NGHP-01 test sample GH116. Individual plots are (A) shear stress (q) vs. effective normal stress (p') on a plane inclined at 45° from the horizontal. Each data point represents the top of a Mohr's circle and together they define a "stress path"; (B) shear stress (q) vs. axial strain, and (C) change in relative pore pressures vs. axial strain.

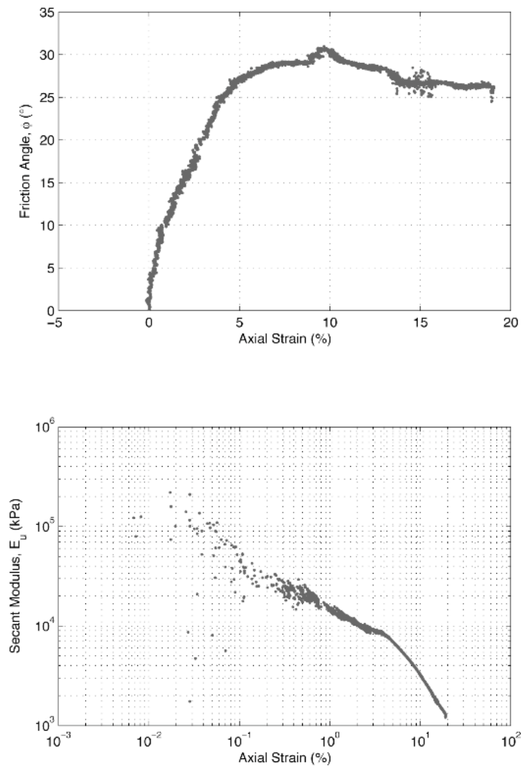


Figure 9. Effective friction angle and secant modulus vs. strain for NGHP-01 sample GH116.

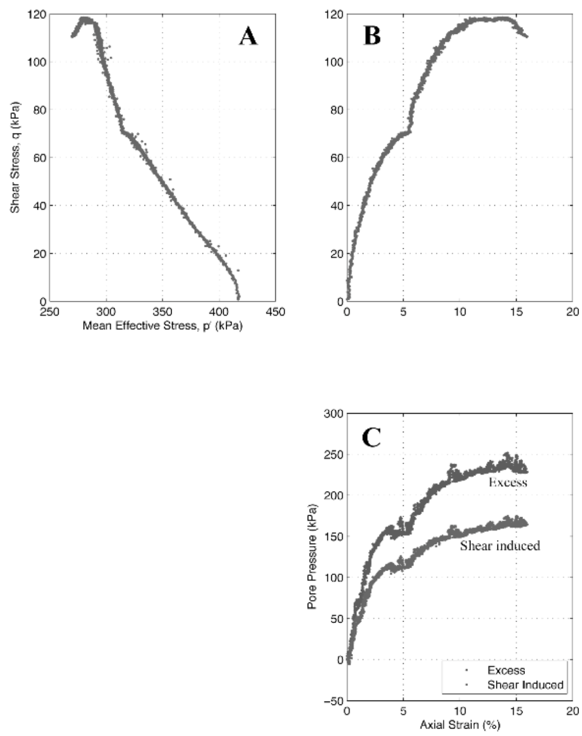
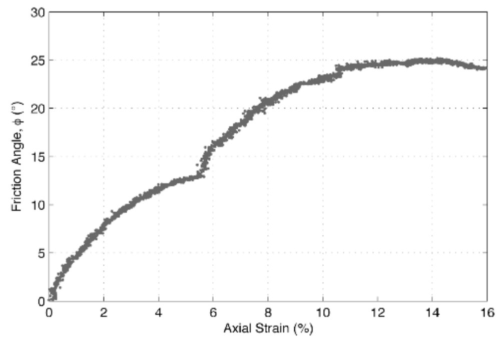


Figure 10. Shear strength results for NGHP-01 test sample GH117. Individual plots are (A) shear

stress (q) vs. effective normal stress (p') on a plane inclined at 45° from the horizontal. Each data point represents the top of a Mohr's circle and together they define a "stress path"; (B) shear stress (q) vs. axial strain, and (C) change in relative pore pressures vs. axial strain.



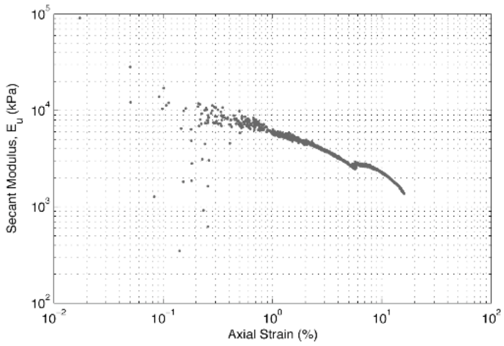


Figure 11. Effective friction angle and secant modulus vs. strain for NGHP-01 sample GH117.

CONCLUSIONS/RECOMMENDATIONS

Obtaining shear strength and other physical properties of hydrate-bearing fine-grained sediment at near in situ effective stress should be accomplished without freezing the sample, and ideally, without depressurizing the material. In these regards, fine-grained marine sediment is more sensitive to handling and transfer techniques than coarse-grained, hydrate-bearing material. This is because freezing and depressurizing coarse-grained, hydrate-rich sediment imparts much less disturbance than freezing fine-grained material.

Applying the same effective stress states makes samples with different disturbance patterns demonstrate similar strength properties. Effective stress exerts a primary control on sediment behavior. Different sediment lithologies and possible pore pressure response over small linear distances makes modeling in situ behavior challenging.

To adequately measure physical properties of gas-hydrate-bearing pressure cores, triaxial test samples must continually be maintained at close to in situ pressure, demonstrating the need to develop a pressure-coring system that performs triaxial testing within the recovery chamber, or a cutting and transfer system that moves the sample into a separate test chamber under in situ pressure.

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