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TESTING OF PRESSURISED CORES CONTAINING GAS HYDRATE FROM DEEP OCEAN SEDIMENTS

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ABSTRACT

The recent development and deployment of HYACINTH and IODP PCS pressure cores on the JOIDES Resolution during Expedition 1 of the Indian National Gas Hydrate Program (NGHP-1) has provided some of the first "undisturbed" samples of gas hydrate in fine grained marine sediments. Some samples, once recovered from the seafloor, were subject to rapid depressurization and subsequent immersion in liquid nitrogen, at approximately -196^{o}C, for use in subsequent laboratory test programs. This paper describes the techniques used at Southampton University, the difficulties encountered, and the results obtained from geotechnical testing of these samples. The original intention had been to pressurize and unfreeze the material before testing it in the Gas Hydrate Resonant Column (GHRC) Apparatus. Initial CT scanning of the samples showed that the sample quality might be too poor for such testing, and this proved to be the case. Instead a suite of geotechnical testing was carried out, the results of which are reported and interpreted in this paper.

Keywords: gas hydrates, pressure cores, geotechnical testing

NOMENCLATURE

DDL	Diffuse double layer
LL	Liquid Limit
PI	Plasticity Index, LL - PL
PL	Plastic Limit
M	The slope of the critical state line on the p′–q plane
p_o'	Isotropic effective stress used to consolidate triaxial specimen
q	deviatoric stress
s_u	Undrained shear strength
w	water content
ϕ'	Effective angle of friction
σ_3	Cell pressure in the triaxial apparatus

INTRODUCTION

The geotechnical properties of hydrate-bearing sediment are of considerable importance, for example in predicting the stability of well-bores drilled in hydrate bearing sediments, and assessing the potential for submarine slope instability as a result either of exploration or development activity, or of environmental change. To date there has been little opportunity to gather such data.

This paper reports a program of laboratory testing carried out on samples obtained using the HYACE (HYdrate Autoclave Coring Equipment)

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pressurized core barrel system, received at Southampton following the Indian National Gas Hydrate Program (NGHP) 01 Expedition [1]. The program comprised a number of stages of testing:

- Initial appraisal of the geometry, disturbance and hydrate content of the frozen cores using CT scanning.
- Creation of a photographic record of the frozen cores following their removal from plastic liners.
- Identification of different sections and masses of core to be used in subsequent testing.
- Testing of the best preserved core in the GHRC.
- Selection of small sub-samples for moisture content, organic content and salinity testing.
- Unfreezing of core, and collection of dissociating gas.
- Imaging of subsamples using scanning electron microscopy.
- Particle size distribution (PSD) testing of subsamples using a Malvern Mastersizer 2000.
- Analysis of subsamples for moisture content, salinity and organic content.
- Combination of samples to provide sufficient mass for subsequent geotechnical testing.
- Geotechnical description of the sediment.
- Plasticity (Atterberg limit) testing at as-received salinity.
- Unconsolidated undrained triaxial shear strength testing at as-received salinity.
- Washing to remove salts.
- Determination of plasticity with zero salinity pore fluid.

Further testing is planned, to determine the effective strength parameters of the material, and the effects of salinity upon these.

INITIAL APPRAISAL AND RECORDING OF THE SAMPLES

Table 1 lists the samples delivered, stored in liquid nitrogen, to the University of Southampton. Both borehole sites were located in the Krishna-Godavari Basin on the Eastern Margin of India [1] within a water depth of 1049 metres below sea level, Holes 10B and 21C were located in relatively close proximity. The five sections of core were divided in order to obtain sub-samples for more detailed testing.

Prior to removal from their plastic core liner and division into sub-samples, full three-dimensional X-ray scans were undertaken using an X-Tek Benchtop CT 160Xi. For this purpose the core was contained in an expanded polystyrene container to limit temperature changes. Initial trials were carried out using frozen clay, instrumented with thermocouples, in order to establish whether there would be sufficient time to carry out the scans before unfreezing and dissociation occurred. These trials showed that a scan period of up to 1 hour could be used with the core section temperature rising from -196°C to around -80°C, thus limiting potential hydrate dissociation.

Figure 1 shows example CT scans of full section core, made whilst frozen and contained within the plastic coreliner. The voiding in the sample can be clearly seen, as can areas of drilling disturbance immediately inside the liner (seen best in the right –hand image). In addition, sheets of steeply-inclined hydrate can be seen.

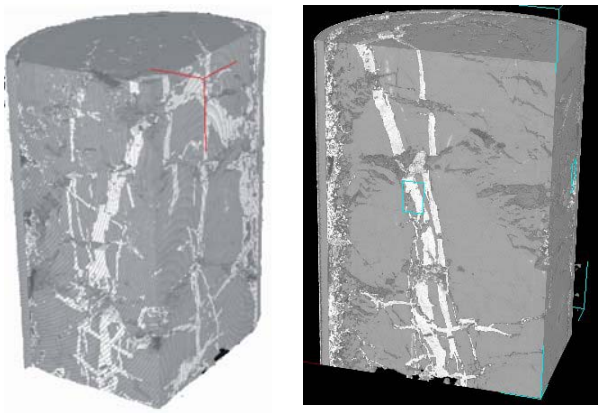

Figure 1. Examples of X-ray CT scans of NGHP-1 samples

Figure 2 shows photographic images of two sections of frozen sample NGHP-1-10B-08Y after their removal from the plastic core liner. The voiding appears to be associated with gas expansion, perhaps as a result of methane coming out of solution in the pore fluid during depressurization and subsequent freezing.

Figure 2. Photographic images of samples after removal from plastic core liner.

Figure 3 shows two images obtained after dissociation, using scanning electron microscopy. Further details and more images can be found in a companion paper by Priest et al. [2].

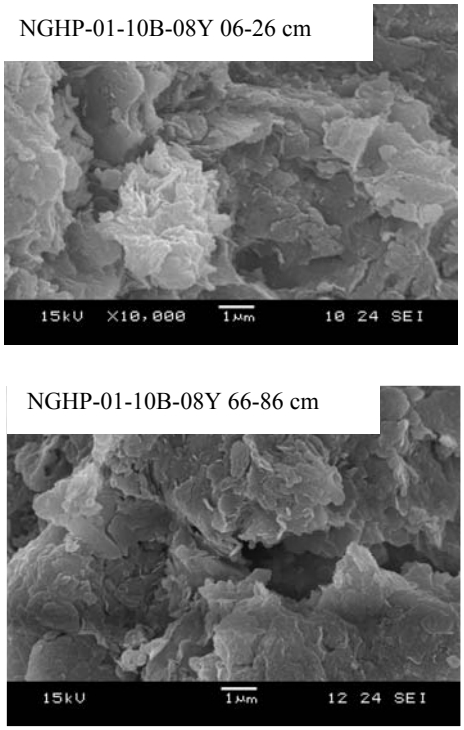

Figure 3. SEM images of dried sediment

Section	Length (cm)	Part	Water content (%)	Total organic content (%)	Chloriide Mmol/kg	Salinity (g/litre)
NGHP-1-10B-08Y (50.1m bsf)	6-26	RCT	64	2.6	634	41
		Part B/C	72	2.5	550	35
	26-46	Part A(i)	68	2.7	689	44
		Part A(ii)	76			
		Part B	69	2.7		40
	46-66	Part A(i)	77	3.0	588	38
		Part A(ii)	75			
		Part B(i)	71	2.6	585	38
		Part B(ii)	77			
		Part C	72	---	---	---
	66-86	Part A(i)	78	2.8	419	27
		Part A(ii)	82	2.8		
		Part A(iii)	81	2.8		
		Part B	81	2.8	---	---
		Part C	78	---	---	---
NGHP-1-21C-02E (56.5m bsf)	23-46	RCT	56	2.1	488	31
		Part B	69	2.8	513	33
Averages			73	2.7		36.7

Table 1. Details of frozen cores received from NGHP-01

GAS COLLECTED DURING DISSOCIATION

The individual sub-samples were dissociated and gas was collected for further analysis. At the time of writing the results are still awaited.

INDEX AND CLASSIFICATION TESTING

Small sub-samples (of the order of 10-20g) were taken from the frozen core. Hydrate veins were avoided, (apart from section 66-86 cm), in an attempt to evaluate the water content and salinity of the host sediment without the effect of the hydrate, which it was thought would increase the water content and freshen the pore water.

The material was subjected to a number of index tests:

- Particle size distribution analysis
- Water content determination
- Pore water salinity
- Organic content.

After initial analyses had been carried out on these sub-samples, the remaining material was unfrozen, and methane was collected for future analysis. The material was then mixed, to provide a single sample for geotechnical sample description, plasticity, specific gravity and strength testing. This material therefore contained additional water released during dissociation of the hydrate.

Geotechnical sample description was carried out to BS5930: 1999 [3], on a sample produced after the detailed testing described below by mixing of all the residual material. The sediment was described as a "Very soft dark grey silty CLAY".

Particle sizing was carried out in a Malvern Mastersizer 2000 using an Autosampler Hydro G. The equipment uses laser diffraction, and determines particle size based on Mie theory [4]. Figure 4 shows particle size distributions determined on subsamples from 34 different sections of core. The material appeared to be uniform in size, and no difference could be seen between the gradings of specimens taken from sample NGHP-1-10B-08Y and those taken from sample NGHP-1-21C-02E. About 20% of the sediment is clay-sized, and 75% is silt-sized, the remainder being sand-sized.

Water contents were determined as a percentage of dry weight, by oven drying in accordance with BS1377: Part 2 [5]. The overall arithmetic mean water content was 73.3%, whilst the arithmetic means of samples NGHP-1-10B-08Y and NGHP-1-21C-02E were 74.7% and 62.9% respectively. These values can be compared with the initial water content of the mixed material, which was found to 75.5%.

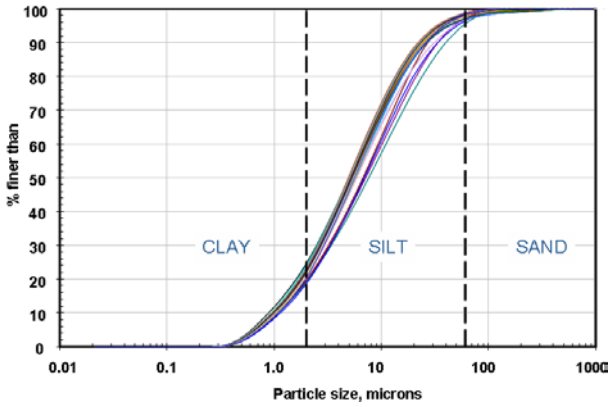

Figure 4. Particle size distributions

Salinity was determined on the basis of chloride ion concentration, using a Dionex ICS2500 ion chromatograph to determine Cl in 9 accurately prepared dilutions of the sub-samples. Single anion and seawater standard solutions were used to calibrate the instrument and verify the accuracy of the determinations. Multiple measurements of one sample solution was undertaken to determine measurement precisions. The results in Table 1 average 36.7%, with chloride ion concentrations varying from 419 to 689 mM. These are somewhat higher values than recorded at the time of core recovery (between 398 and 634 mM [1]), and have a slightly higher average than seawater.

Plasticity was determined using the cone method (BS 1377: Part 2:1990 [5] clause 4.3) for the liquid limit. Salinity determinations (see above) had indicated levels as high, or in some cases somewhat higher than sea water. Increasing salinity increases the thickness of the diffuse double layer on clay colloids, and because absorbed water is not free, an increase in salinity leads to an increase in strength at any given water content. It is routine practice when testing sediments which do not have saline pore water to use tap water to increase the moisture content

when determining the liquid limit, but this would have given false values in this case.

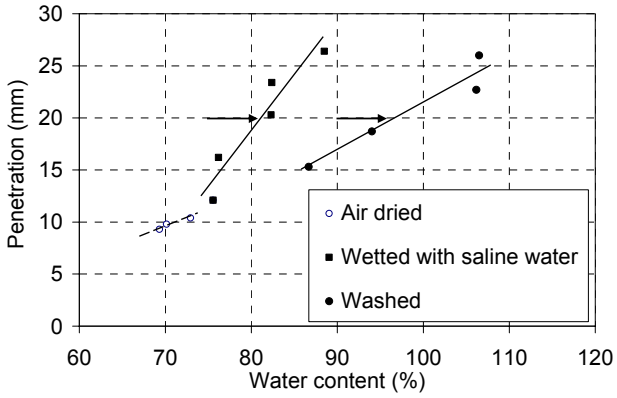

Figure 5. Penetration of liquid limit cone as a function of moisture content

Figure 5 shows cone penetration as a function of water content. The starting water content was 75.5%. Lower moisture contents were obtained by air drying, whilst higher values were achieved by adding water with a salinity of 35g/litre. The sample was initially somewhat dry of the liquid limit, which was found (at 20mm penetration) to be 81%. The plastic limit was found after air drying to be 33%

In a further test, material was air dried, ground down using a mortar and pestle, and then washed in distilled water to remove its salinity. The liquid and plastic limits were then found to be 96% (see Figure 5) and 33% respectively.

The **specific gravity** (grain density) of the combined sediment sample was determined using the 50ml density bottle method (BS1377:Part 2: 1990 [5] clause 8.3) to be 2.74. Grain densities measured during the cruise [1] varied from 2.44 to 2.86 g/cm^3, with an average of 2.71g/cm^3. Higher laboratory values of grain density are likely to be the more accurate, since they will reflect more effective de-airing [6].

Organic contents of a number of sub-samples were determined as shown in Table 1. The arithmetic mean Total Organic Content was found to be 2.7%.

STRENGTH TESTING

The liquid limit cone penetration values can be used to obtain a measure of strength under high rates of loading, but it is preferable to carry out testing under the more controlled conditions provided by the standard unconsolidated undrained triaxial compression test (BS1377:Part7 [7] clause 8), where failure occurs in a period of between 5-15 minutes.

The combined sample was tested using this method, using a 38mm diameter by 76mm high specimen under a machine rate of strain of 2% / minute. At its initial moisture content of 75.5% the undrained shear strength was found to be only 6.2 kPa (cf. the strength at the liquid limit, which is of the order of 2 kPa [8], and the plastic limit, where the strength is about 170 kPa [9]. Forming and mounting triaxial specimens at low shear strengths such as this is a challenge, which explains why attempts to carry out resonant column testing were unsuccessful.

Samples at lower moisture content and higher strengths were obtained by consolidating the combined sample under cell pressures of 100kPa and 200kPa, with atmospheric back pressure, giving undrained triaxial shear strengths of 26 kPa and 98 kPa respectively. The first sample was consolidated in the triaxial cell; at higher effective stresses the sample was consolidated in a hydraulic oedometer, and was then tube sampled to obtain a 38mm diameter specimen used in the triaxial strength test. The increase in shear strength with effective consolidation pressure is higher than would normally be expected for a terrestrial sediment with fresh pore water.

INTERPRETATION OF RESULTS

The development of a testing program was a challenge, given that the state of the core (which was frozen and concealed within plastic core liner) was unknown, and that it was estimated that less than 2kg of material would be available.

The results obtained from this small but detailed study of hydrate-bearing core have been compared with values from logging while drilling, and values in the literature, in relation to water content / porosity, undrained shear strength, and effective angle of friction.

Heterogeneity

Testing of sub-samples indicated that the sediment did not vary significantly from section to section. For example, particle size distributions (even though based upon samples of only a gram or so) were uniform (Figure 4) and organic contents were low. This suggests an absence of fabric (in the sense used by Rowe [10]). Water content and salinity varied to a greater extent, but this is to be expected given the spatial variation of hydrate content, for example as ca be seen in Figure 1.

Water content

The moisture content range of the sub-samples tested in the laboratory (63-75%) is within the range of WCs reported below 10m bsf (46-81%) in Figure F26 of Collett et al. [1]. The generally higher values may reflect the contribution of water from dissociating hydrate, the existence of which is shown by the low values of bulk density at approximately 30 (1.46g/cm^3), 41 (1.37 g/cm^3), 50 (1.24 g/cm^3) and 59m bsf (1.43 g/cm^3) in the Logging While Drilling (LWD (RHOB)) data. A bulk density of 1.4g/cm^3 implies a water content of approximately 120%.

Salinity values of subsamples are compared with their moisture contents in Figure 6. The curve showing the calculated effect of added fresh water (for example from dissociating hydrate) is plotted for an initial water content of 70% and a salinity of 38%.

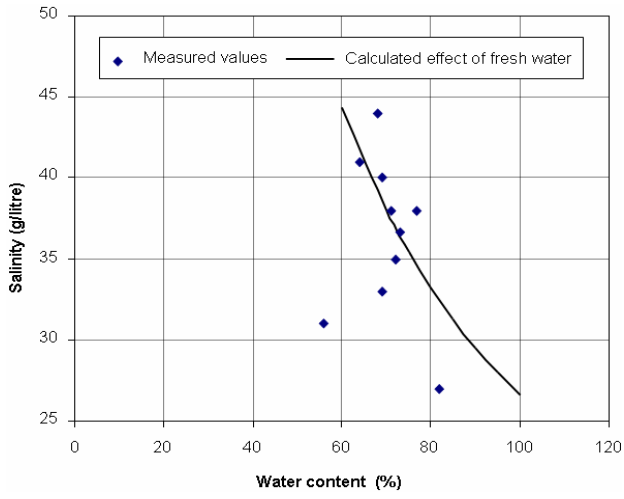

Figure 6. Salinity as a function of water content.

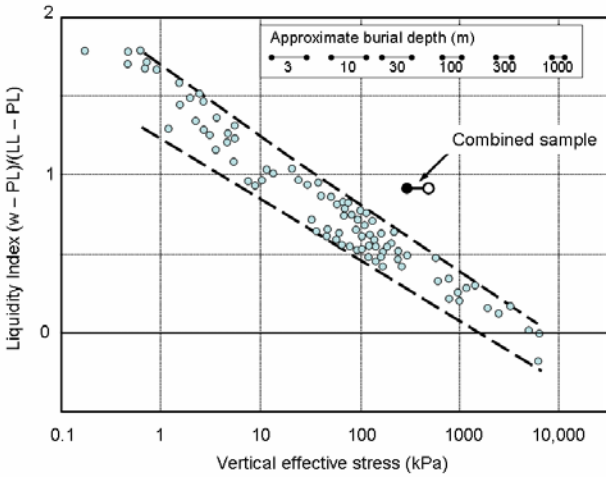

Figure 7. Comparison of Liquidity Index of combined sample with data from Skempton [11]

On the basis of the field LWD (RHOB) measurements the vertical effective stress was estimated from Figure F26 of Collett et al. [1] for NHP-1 Hole 10B to be 280kPa at 50m bsf. Figure 7 shows the Liquidity Index of the sample, and compares it with Skempton's [11] data. It shows that the porosity of the sediment is far higher than would be expected at the vertical effective stress to which it was subjected before sampling.

Undrained shear strength

Undrained shear strength is shown as a function of water content, for the combined sample tested at its as-received salinity (approximately that of sea water). Data from triaxial tests are combined with estimated strengths at the Liquid and Plastic Limits (Figure 8).

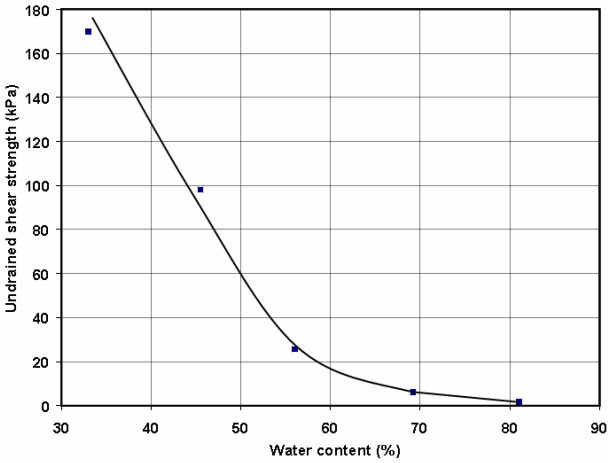

Figure 8. Undrained shear strength v. water content for the combined sample.

Chandler [12] has shown the variation of K_o consolidated undrained shear strength to vertical effective stress for reconstituted clays to vary between 0.23 and 0.46, and typically to be about 0.34. On this basis, the remolded undrained shear strength of the sediment at 50m bsf would be expected to be of the order of 95kPa ±30kPa. The measured shear strength (6.2kPa) falls very far below these values, perhaps as a result of the release of hydrate-held water into the sediment during dissociation, or perhaps as the result of mechanical destructuring during remolding.

The cause of the unusually high Liquidity Index and associated low undrained shear strength is unknown, but a number of factors may have contributed to the measurements reported here:

- Firstly, the moisture contents of the samples may have been raised by the contribution of water freed during hydrate dissociation,
- Secondly, the sediment was in all probability structured *in situ*, so that it could support the weight of overburden. The process of sampling and dissociation will have destructured it (Clayton et al.) [13],
- Thirdly, changes in pore water salinity during dissociation may have had an effect in reducing the undrained shear strength, and finally
- The relationships reported by Skempton [11] may not be appropriate for this young marine clay and its saline pore water.

The first hypothesis does not seem to be supported by our data. Figure 8 suggests that a moisture content increase of around 30% would be required to reduce the undrained strength from the expected value (under an effective overburden of 280kPa) of 95kPa to the measured value of 6kPa, whilst the trend line in Figure 6 suggests that this should have reduced salinity to about 27%.

It is clear from Figure 1 and from the CT images presented in a companion paper [2] that the sediment had macrostructure *in situ*, in the form of cross-cutting hydrate sheets. If hydrate formed before the application of significant overburden pressure then it would seem possible that it could support the sediment, allowing it to remain at a high porosity. Destructuring during sampling may therefore be giving a false view of the in-situ undisturbed strength of the sediment.

Changes in pore water salinity undoubtedly have an effect on the undrained strength – moisture content relationship of clayey sediment. All other things being equal, a decrease in pore fluid salinity leads to an increase in the thickness of the diffuse double layer (DDL), and hence to an increase in the measured Liquid Limit (Figure 5), in this case by some 15%. This effect has also been observed by van Paassen and Gareau [14]. For the same water content this has the effect of increasing the undrained shear strength of a remolded sediment. In contrast Skempton and Northey [15] and Bjerrum and Rosenqvist [16] show for Norwegian clays that leaching has the effect of reducing the thickness of the DDL, and reducing the Liquid Limit.

The observed effect of increased salinity increasing the remolded undrained shear strength of clay at a given water content (Rosenqvist [17]) is presumably a result of the effect of pore water chemistry promoting an open edge-to-face flocculated fabric. The effect of pore fluid chemistry on the sedimentation process is complex, but it appears possible that high pore fluid salinity may lead to an apparently under-consolidated soil, as suggested for sediment recovered from the Caspian Sea (van Paassen and Gareau [13]).

Effective angle of friction

For the specimens consolidated against atmospheric back pressure the mean effective stress before shearing, p_o', is equal to the cell pressure during consolidation, σ_3. This allows an estimate of the effective angle of friction of the soil, since from critical state theory (Roscoe and Burland [18] at failure, for Modified Cam Clay $p' = 0.5\ p_o'$. For Cam Clay the ratio is 1/e, or 0.368 (Schofield and Wroth [19]). Thus at the critical state, and since $q = 2.su$

$$M = q/0.5p_o' = 4.s_u / p_o' \text{ (for Modified Cam Clay)}$$

or

$$M = q/0.368p_o' = 5.4.s_u / p_o' \text{ (for Cam Clay)}$$

and since

$$M = 6.\ \sin\phi'/(3-\sin\phi')$$

an estimate of the effective angle of friction, ϕ', can be obtained. For an isotropic effective stress of 100kPa values of $\phi' = 26^o$ and 34^o ($c' = 0$) are obtained. These values seem somewhat high for a clay with a Plasticity Index of 48% (for example see Kenney [20], whose data suggests an effective angle of friction at this plasticity of between 20^o and 30^o) but may be a function of the salinity of the pore water. Values calculated from the specimen consolidated at 200kPa appeared unreasonably high.

Deposition of sediment under K_o conditions implies the ability to support shear stress, i.e. the difference between the vertical and horizontal effective stress. Using Jaky's equation for K_o and an effective angle of friction of 30^o with the previously estimated value of vertical effective stress, for example, the deviatoric stress at 50m bsf is of the order of 140kPa, and requires a normally consolidated sediment to have a minimum undrained shear strength of 70kPa, or about 10 times the value measured on the combined sample.

CONCLUSIONS

A program of geotechnical laboratory tests has been carried out on hydrate-bearing sediment recovered from more than 1000m below sea level, and approximately 50m below sea floor, using a pressurized corebarrel. Despite the fact that less than 2kg of sample was made available, significant testing was possible.

The sediment was a clay of high plasticity, with a Liquid Limit of 81% and a Plasticity Index of 48%. Test results showed that the sample had maintained a high salinity despite having a water content far in excess of that expected for a material of this plasticity under an effective overburden pressure of almost 300kPa. A portion of the combined sample was washed to remove its salinity, and the liquid limit was found to increase by 15%.

The effect of the high water content was that the sample had a very low remolded undrained shear, of the order of only 6kPa. This, and the voiding caused by methane dissolution during depressurizing and before freezing, made specimen preparation for advanced geotechnical laboratory testing impossible.

It is suggested that the apparent under-consolidation of the material may have resulted either from the salinity of its pore fluid, or from the presence of the many hydrate veins that were observed in the core during CT scanning. A proper evaluation of the mechanical behavior of hydrate-bearing clay will require the future development of sample preparation and testing protocols that avoid depressurizing samples before they are frozen.

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