NATIONAL ENERGY TECHNOLOGY LABORATORY
OIL & GAS

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Quarterly Research Performance Progress Report (Period Ending 9/30/2018)

A multi-scale experimental investigation of flow properties in coarse-grained hydrate reservoirs during production

Project Period (10/1/2016-9/30/2019)

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Office of Fossil Energy

DISCLAIMER

1. ACCOMPLISHMENTS:

What was done? What was learned?

This report outlines the progress of the fourth quarter of the second fiscal year in the second budget period. Highlights from the period include:

- Pore-Scale experiments show preferential hydrate formation in coarser-grained sediment:

  New Micro-Raman experiments generated hydrate in a heterogeneous packing of two segments of sediment: one, GOM2 Lithofacies 2 (mass medium diameter ~50 μm), and the other, a coarser-grained clay-free natural quartz sand (210 μm to 297 μm). Results shows that although a large number of hydrate nucleation sites initiated simultaneously in both sediments, over time a higher concentration of methane hydrate formed in the coarser sand as a result of the lower capillary force present compared to Lithofacies 2. This result is consistent with reservoir scale models showing higher hydrate build up in areas of lower capillary pressure and provides insight into the naturally occurring higher hydrate concentration in GC 955 H005 Lithofacies 2 as compared to the finer interbedded layers of the clayey-silt Lithofacies 3. Details of the experiment and results can be found under Task 9.

- Core-Scale Experiments measure steady-state permeability in the presence of methane hydrate for the first time:

  Methane and brine co-injection experiments were performed at a hydrate saturation of 25% and the relative permeabilities of each core section were measured. We believe these are the first ever measurements of relative permeability at steady state with simultaneous flow of gas and brine in the presence of hydrate. Details of the experiment and results can be found under Task 6. Table 6.1 shows the calculated permeability values based on the slope of pressure drop versus flow rate at a 50:50 methane:brine injection ratio by volume, and Table 6.2 shows a calculated permeability value for a 90:10 methane:brine injection ratio by volume.

A. What are the major goals of the project?

The goals of this project are to provide a systematic understanding of permeability, relative permeability and dissipation behavior in coarse-grained methane hydrate - sediment reservoirs. The results will inform reservoir simulation efforts, which will be critical to determining the viability of the coarse-grained hydrate reservoir as an energy resource. We will perform our investigation at the macro- (core) and micro- (pore) scale.

At the macro- (core) scale, we will: 1) measure the relative permeability of the hydrate reservoir to gas and water flow in the presence of hydrate at various pore saturations; and 2) depressurize the hydrate reservoir at a range of initial saturations to observe mass transport and at what time scale local equilibrium describes disassociation behavior. Simultaneously, at the micro (pore) scale, we will 1) use micro-CT to observe the habit of the hydrate, gas, and water phases within the pore space at a range of initial saturations and then image the evolution of these habits during dissociation, and 2) use optical micro-Raman Spectroscopy to images phases and molecules/salinity present both at initial saturations and at stages of dissociation. We will use our micro-scale observations to inform our macro-scale observations of relative permeability and dissipation behavior.

In Phase 1, we first demonstrated our ability to systematically manufacture sand-pack hydrate samples at a range of hydrate saturations. We then measured the permeability of the hydrate-saturated sand pack to flow a single brine phase and depressurized the hydrate-saturated sand packs and observed the kinetic (time-dependent) behavior. Simultaneously we built a micro-CT pressure container and a micro-Raman Spectroscopy chamber and imaged the pore-scale habit, phases, and pore fluid chemistry of sand-pack hydrate samples. We then made observations on our hydrate-saturated sand-packs.

In Phase 2, we will measure relative permeability to water and gas in the presence of hydrate in sand-packs using co-injection of water and gas. We will also extend our measurements from sand-pack models of hydrate to observations of actual Gulf of Mexico material. We will also measure relative permeability in intact samples to be recovered from the upcoming Gulf of Mexico 2017 hydrate coring expedition. We will also perform dissipation experiments on intact Gulf of Mexico pressure cores. At the micro-scale we will perform micro-Raman and micro-Ct imaging on hydrate samples composed from Gulf of Mexico sediment.

The Project Milestones are listed in the table below.

Milestone Description	Planned Completion	Actual Completion	Verification Method	Comments
Milestone 1.A: Project Kick-off Meeting	11/22/2016 (Y1Q1)	11/22/16	Presentation	Complete
Milestone 1.B: Achieve hydrate formation in sand-pack_Task_2.0_Macro-Scale:	6/27/2017 (Y1Q3)	8/11/17	Documentation of milestone achievement within required project reporting / deliverables (Deliverable 2.1)	Complete, *Documentation in the Y1Q3 quarterly and Phase 1 report*
Milestone 1.C: Controlled and measured hydrate saturation using different methods_Task_2.0_Macro-Scale:_1	3/27/2018 (Y2Q2)	3/27/18	Documentation of milestone achievement within required project reporting / deliverables (Deliverable 2.1)	Complete, *Documentation in Y2Q2 quarterly and Phase 1 report*
3 Milestone 1.D: Achieved depressurization and demonstrated mass balance_Task_3.0_Macro-Scale:	3/27/2018 (Y2Q2)	12/18/2017	Documentation of milestone achievement within required project reporting / deliverables (Deliverable 3.1)	Complete, *Documentation in the Y2Q1 quarterly and Phase 1 report*
Milestone 1.E: Built and tested micro-consolidation device_Task_4.0_Micro-Scale:_1	6/27/2017 (Y1Q3)	6/27/2017	Documentation of milestone achievement within required project reporting / deliverables (Deliverable 4.1)	Complete, *Documentation in Y1Q3 quarterly and Phase 1 report*
Milestone 1.F: Achieved Hydrate formation and measurements in Micro-CT consolidation device_Task_4.0_Micro-Scale:_1	3/27/2018 (Y2Q2)	2/15/18	Documentation of milestone achievement within required project reporting / deliverables (Deliverable 4.1)	Complete, *Documentation in Y2Q2 quarterly and Phase 1 report*
Milestone 1.G: Built and integrated high-pressure gas	3/27/2018 (Y2Q2)	6/27/17	Documentation of milestone achievement within required	Complete,

mixing chamber Task 5.0 Micro-Scale:			project reporting / deliverables (Deliverable 5.1)	*Documentation in Y1Q3 quarterly and Phase 1 report*
Milestone 1.H: Micro-Raman analysis of synthetic complex methane hydrate Task 5.0 Micro-Scale:	3/28/2018 (Y2Q2)	3/27/18	Documentation of milestone achievement within required project reporting / deliverables (Deliverable 5.1)	Complete, *Documentation in Y2Q2 quarterly and Phase 1 report*
Milestone 2.A - Measurement of relative permeability in sand-pack cores. (See Subtask 6.1) Task 6.0 Macro-Scale: 2 Task 6.0 Macro -Scale: 2	1/17/2019 (Y3Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 6.1)	In progress
Milestone 2.B - Measurement of relative permeability in intact pressure cores. (See Subtask 6.2) Task 6.0 Macro-Scale: 2 Task 6.0 Macro -Scale: 2	9/30/2019 (Y3Q4)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 6.1)	
Milestone 2.C -Depressurization of intact hydrate samples and documentation of thermodynamic behavior. (See Subtask 7.1 and 7.2) Task 7.0 Macro-Scale: Task 7.0 Macro-Scale:	9/30/2019 (Y3Q4)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 7.1)	In progress
Milestone 2.D - Achieved gas production from GOM^2 samples monitored by micro-CT. (See Subtask 8.1 and 8.2) Task 8.0 Micro-Scale: Task 8.0 Micro-Scale:	9/30/2019 (Y3Q4)		Documentation of milestone achievement within required project reporting / deliverables Report (Deliverable 8.1)	In progress
Milestone 2.E - Building a chamber to prepare natural samples for 2D-3D micro-Raman analysis; (See Subtask 9.1 and 9.2) Task 9.0 Micro-Scale: Task 9.0 Micro-Scale:	1/17/2019 (Y3Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 9.1)	In progress
Milestone 2.F - 2D micro-Raman analysis of natural methane hydrate samples at depressurization; (See Subtask 9.1 and 9.2) Task 9.0 Micro-Scale: Task 9.0 Micro-Scale: 1	9/30/2019 (Y3Q4)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 9.1)	In progress

B. What was accomplished under these goals?

PAST- BUDGET PERIOD 1

Task 1.0 Project Management and Planning

Planned Finish: 09/30/19
Actual Finish: In progress continued in Phase 2, see Task 1 below.

Task 2.0 Macro-Scale: Relative Permeability of Methane Hydrate Sand Packs

Subtask 2.1 Laboratory Creation of Sand-Pack Samples at Varying Hydrate Levels
Planned Finish: 6/ 27/17
Actual Finish: 8/11/17 Complete

Documentation of subtask completion in Y1Q4 Quarterly and the Phase 1 report per the SOPO (Deliverable 2.1).

Subtask 2.2 Steady-State Permeability of Gas and Water of Sand-Pack Hydrate Samples
Planned Finish: 3/27/18
Actual Finish: Complete

Documentation of subtask completion in Y2Q2 Quarterly and the Phase 1 report per the SOPO (Deliverable 2.1).

Task 3.0 Macro-Scale: Depressurization of Methane Hydrate Sand Packs

Subtask 3.1 Depressurization Tests
Planned Finish: 6/27/17
Actual Finish: 3/27/2018 Complete

Documentation of subtask completion in was made in the Phase 1 report per the SOPO (Deliverable 3.1).

Subtask 3.2 Depressurization Tests with CAT scan
Planned Finish: 03/27/18
Actual Finish: 3/27/2018 Complete

Documentation of subtask completion in was made in the Phase 1 report per the SOPO (Deliverable 3.1).

Task 4.0 Micro-Scale: CT Observation of Methane Hydrate Sand Packs

Subtask 4.1 Design and Build a Micro-CT compatible Pressure Vessel
Planned Finish: 6/27/17
Actual Finish: 6/27/2017 Complete

Subtask 4.2 Micro-Scale CT Observations and Analysis
Planned Finish: 03/27/18
Actual Finish: 2/15/2018 Complete

Documentation of Milestone 1.F was included in the Y2 Q2 report and the Phase 1 report per the SOPO (Deliverable 4.1)

Task 5.0 Micro-Scale: Raman Observation of Methane-Gas-Water Systems

Subtask 5.1 Design and Build a Micro-Raman compatible Pressure Vessel
Planned Finish: 6/27/17
Actual Finish: 6/27/17 Complete

Documentation of subtask completion in Y1Q3 Quarterly, Documentation of Milestone 1.G included in the Phase 1 report per the SOPO (Deliverable 5.1)

Subtask 5.2 Micro-scale petrochemistry
Planned Finish: 03/31/18
Actual Finish: 03/27/2018 Complete

Documentation of Milestone 1.H included in the Y2Q2 and Phase 1 report per the SOPO (Deliverable 5.1)

Subtask 5.3 Diffusion kinetics of methane release
Planned Finish: 3/27/18
Actual Finish: 3/27/2018

Documentation of Milestone 1.H included in the Y2Q2 and Phase 1 report per the SOPO (Deliverable 5.1)

Decision Point: Budget Period 2 Continuation

Continuation Application submitted on March 5. Continuation approved March 26, 2018.

CURRENT – BUDGET PERIOD 2

Task 1.0 Project Management and Planning

Planned Finish: 09/30/19
Actual Finish: In progress

This tasks continues from Phase 1.
The seventh Quarter Report was submitted on August 1, 2018.

Link to actions for next Quarter, Task 1

Task 6.0 Macro-Scale: Relative Permeability of Methane Hydrate Sand Packs and Intact Pressure Core Samples

Subtask 6.1 Steady-State Relative Permeability Measurements of Sand-Pack Hydrate Samples
Planned Finish: 1/17/19
Actual Finish: In Progress

The tasks for this quarter were to continue performing relative permeability experiments for simultaneous flow of gas and brine at a range of hydrate saturations. A total of 3 experiments were performed on a Boise sandstone core at 25% hydrate saturation. Before the experiments, methane hydrate was formed in the pore space of the sandstone core through the excess gas method. The core was first saturated with water to a 35% water saturation. Next, an Isco pump filled with methane was connected to the core, and a constant pressure of 1250 psi was applied. As hydrate formed, the Isco pump injected more methane to maintain the pressure of 1250 psi. We monitored the volume of methane injected until the hydrate saturation reached 25%. All 3 tests were run on this core at varying gas and brine flow rates.

Results

Experiment 1: 50:50 methane:brine by volume co-injection

This experiment was performed with a confining pressure of 1700 psi and a pore pressure of 1250 psi, and differential pressure was measured using five differential pressure transducers located at 4-inch intervals along the core (Figure 6.1). Brine was injected using a Quizix pump, and methane was injected using a Bronkhorst mass flow controller. The temperature inside the core was 6 ± 1°C, and the fluids injected during this test were 9.8 wt% salinity brine and methane. The injection ratios were volumetric ratios of the fluids at the pressure and temperature conditions inside the core. Methane and brine co-injection tests were first measured as a function of pore volumes injected, with flow rates of 2.5, 5, 7.5, and 10 mL/min. At each flow rate, steady state was reached quickly as indicated by the relatively constant values of dP. However, as more pore volumes were injected, the differential pressures of Section 1 and Section 2 began to decrease above injection rates of 5 ml/min (Figure 6.2), potentially due to hydrate dissociation or movement.

Despite this phenomenon, the relative permeabilities of each section were still measured. The differential pressure at steady state for each flow rate was plotted, and linear trends for each section were established. The viscosity value used for methane was 0.0129 cp, and the viscosity value used for brine was 1.45 cp. The differential pressures at flow rates above 5 ml/min in Section 1 and above 7.5 ml/min in Section 2 were ignored in establishing the linear trend lines (Figure 6.2). Effective permeability for each phase was then calculated using the slope of the trend line from the flow rate and pressure values in Figure 6.3 and Darcy's Law. Relative permeability was then calculated by dividing the effective phase permeability by the absolute permeability of the rock. This absolute permeability value was calculated during a previous experiment in May 2018. The permeability values from the 50:50 methane:brine volume ratio injection experiment are presented in Table 6.1.

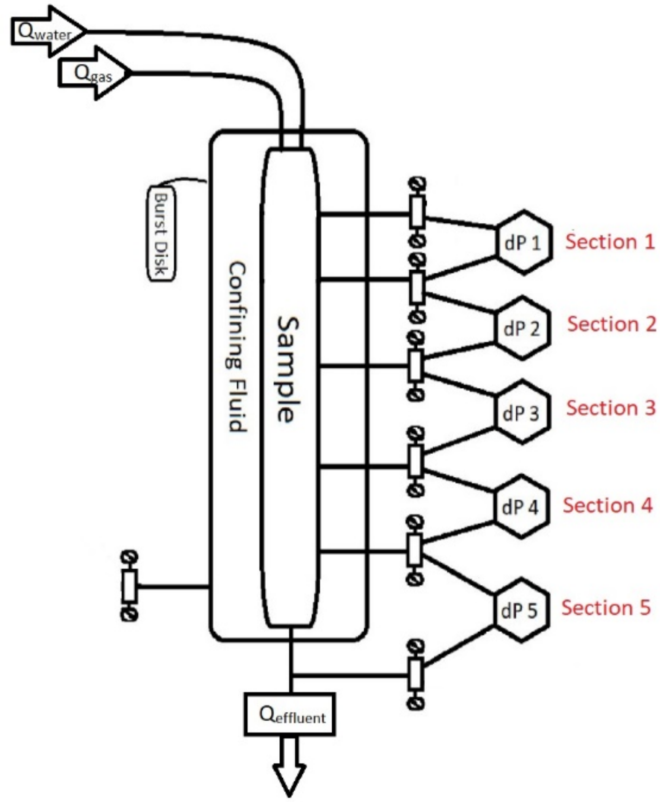

Figure 6.1. Schematic of core holder showing location of pressure measurements.

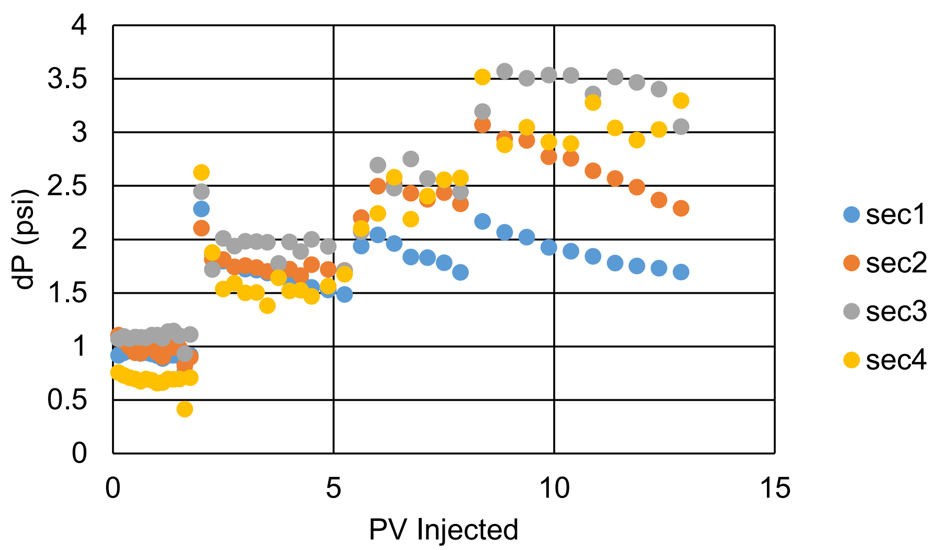

Figure 6.2. Pressure drop versus pore volumes (PV) injected for Experiment 1 at a 50:50 methane:brine ratio by volume. The different colors refer to measurements across different sections of the core (cf. Figure 6.1). Each step change in dP corresponds to an increase in flow rate.

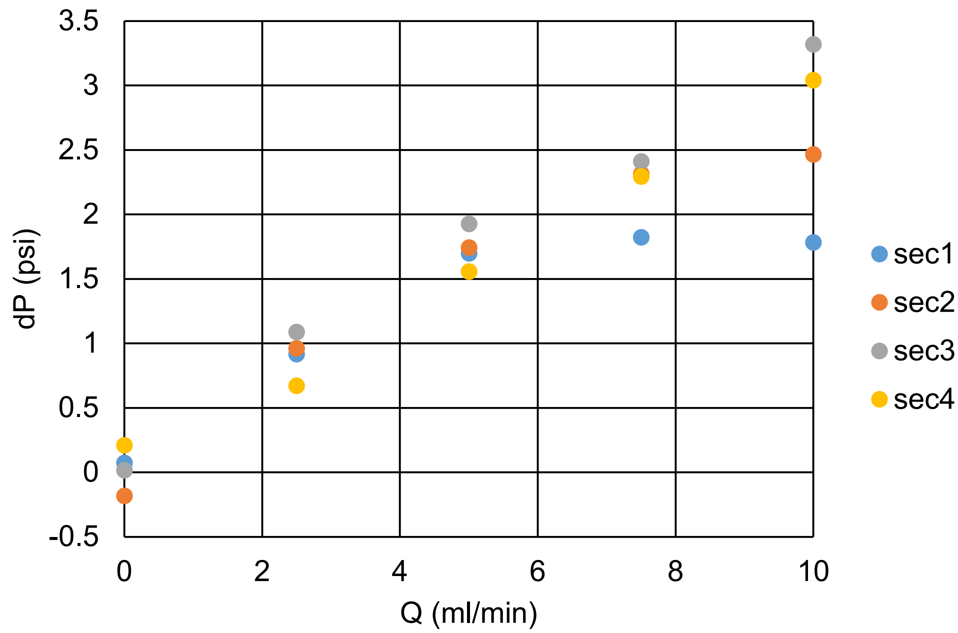

Figure 6.3. Pressure drop versus flow rate (Q) for Experiment 1 at 50:50 methane:brine injection ratio by volume. Different colors correspond to different sections of the core (cf. Figure 6.1).

Table 6.1. Calculated permeability values for Experiment 1 based on the slope of dP vs. Q at a 50:50 methane:brine injection ratio by volume.

Section	Effective Brine Phase Permeability, Darcy	Brine Relative Permeability	Effective Methane Phase Permeability, Darcy	Methane Relative Permeability
1	0.67	0.13	0.0085	0.0017
2	0.57	0.15	0.0071	0.0018
3	0.69	0.13	0.0087	0.0016
4	0.75	0.16	0.0094	0.0020

Experiment 2: 90:10 methane:brine by volume co-injection at single flow rate

A co-injection experiment of methane and brine at a 90:10 ratio was also performed at a hydrate saturation of 25% formed using the excess gas method. To determine the point at which steady state was reached, 175 pore volumes were flowed through the core at a brine flow rate of 1 mL/min. At this ratio, steady state appeared to occur at around 50 pore volumes injected (Figure 6.4).

Experiment 3: 90:10 methane:brine by volume co-injection at variable flow rates

This experiment was performed following the steady-state test at a 90:10 methane:brine injection ratio by volume, and was also performed at a 25% hydrate saturation. Co-injection of methane and brine at different flow rates was tested, but a linear relationship between dP and Q was only achieved for one section, section 3 (Figure 6.5). We used to the same viscosity parameters to calculate permeability for this experiment as were used in Experiment 1. Permeability values from this experiment are presented in Table 6.2. The relative permeability of methane gas is fairly low.

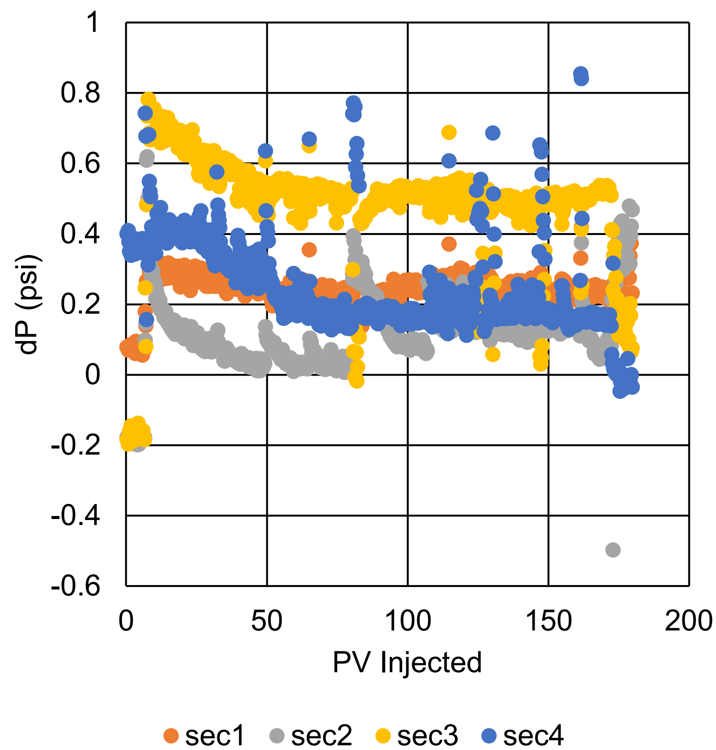

Figure 6.4. Pressure drop versus pore volumes (PV) injected during Experiment 2 at a 90:10 methane:brine injection ratio by volume.

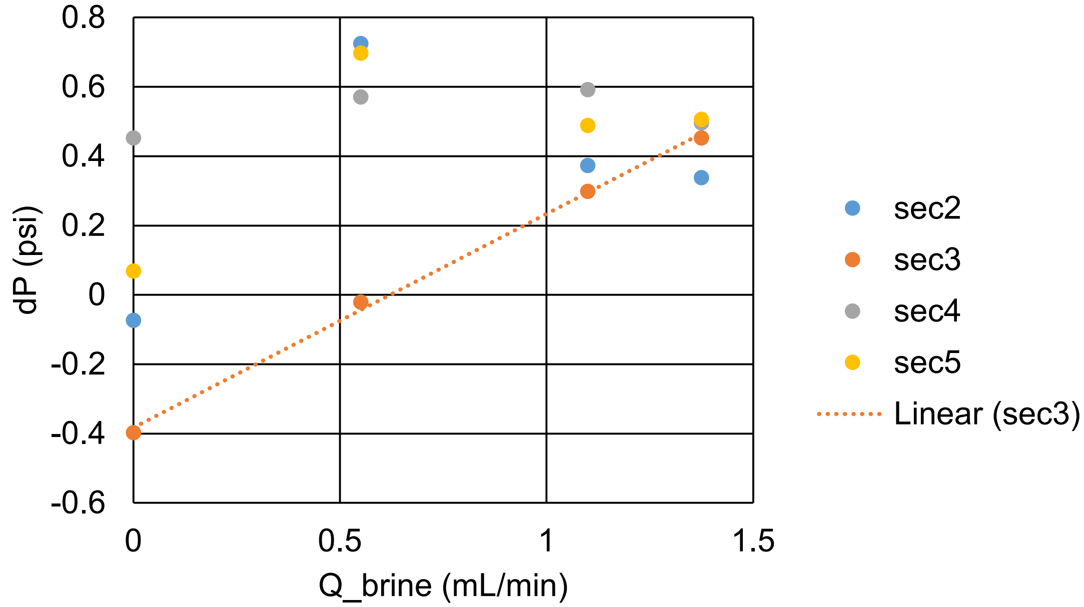

Figure 6.5. Pressure drop versus brine flow rate (Q_brine) during Experiment 3 at a 90:10 methane:brine injection ratio by volume. Linearity was only observed in Section 3.

Table 6.2. Permeability values for 90:10 methane:brine injection ratio by volume during Experiment 3.

Section	Effective Brine Phase Permeability, Darcy	Brine Relative Permeability	Effective Methane Phase Permeability, Darcy	Methane Relative Permeability
3	0.353	0.064	0.0402	0.0073

Link to actions for next Quarter, Task 6

Subtask 6.2 Steady-State Relative Permeability Measurements of Intact Pressure Cores
Planned Finish: 9/30/19
Actual Finish: Not Started

Task 7.0 Macro-Scale: Depressurization of Methane Hydrate Sand Packs and Intact Pressure Core Samples

Subtask 7.1 Depressurization of sand-pack hydrate samples
Planned Finish: 1/17/19
Actual Finish: In Progress

We did not run any depressurization of sand-pack hydrate samples during Q3. We began to prepare sand pack samples to be formed during Q4 using the excess gas method of Task 2.0. The goal of this work is to observe dissociation behavior across multiple formation methods and a larger range in hydrate saturations.

We are working to revise and resubmit a manuscript based on our depressurization experiments from Task 3.0 in sand packs containing hydrate formed with a gas injection method. These results highlight (1) the ability to estimate the sample salinity by monitoring the initial pressure of hydrate dissociation, (2) the deviation of observed pressure during dissociation from the pressure predicted by homogenous conditions, and (3) influence of salt diffusion on the form pressure rebounds. These results show that when hydrate dissociation begins, localized freshening and cooling around the hydrate sets up salinity and heat gradients that change the conditions around the dissociating hydrate.

Link to actions for next Quarter, Task 7

Subtask 7.2 Depressurization of intact pressure cores
Planned Finish: 9/30/19
Actual Finish: In Progress

We depressurized 1 core section recovered from the northern Gulf of Mexico Green Canyon 955 during UT-GOM2-1. This sample contained both sandy silt and silty sand lithofacies, with an expected mix of high and low hydrate saturations. During this dissociation, we allowed for recovery and monitoring of pressure between degassing steps. We calculate a hydrate saturation of 44% of the pore volume. Based on the pressure and temperature of the initial dissociation we estimate an in situ salinity of the sample between 30 to 36 parts per thousand (near seawater concentration). To this point, we have depressurized compromised cores (cores that partially lost pressure and were possibly damaged during processing) to establish our depressurization method and will move on to degassing lithofacies-specific samples in non-compromised cores during Q4.

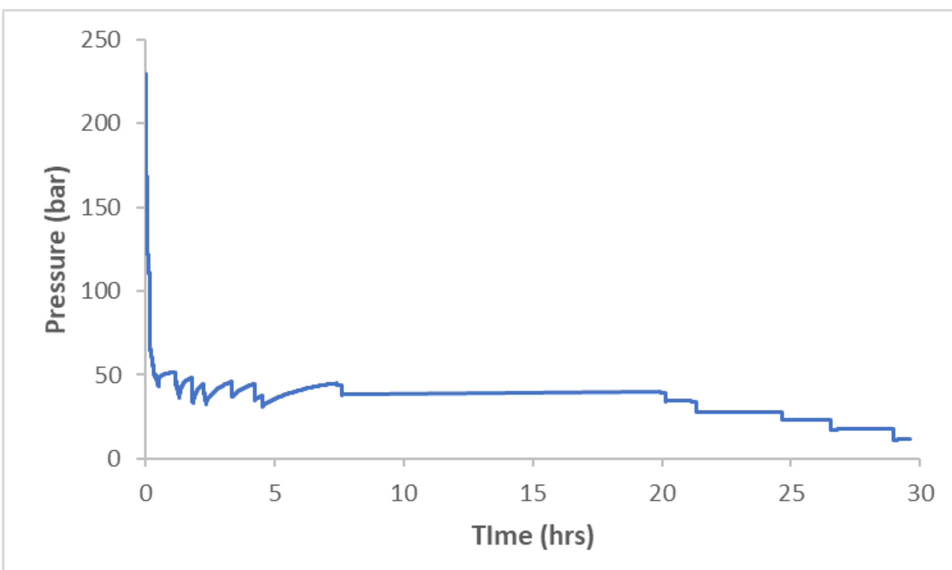

Figure 7.1. Sample pressure during for section H005-6FB-2, 21.5-42.5 cm, a multiple-lithofacies and compromised core. Note the pressure rebounds that occur after each degassing step during dissociation.

Link to actions for next Quarter, Task 7

Task 8.0 Micro-Scale: CT experiments on Gulf of Mexico Sand Packs

Subtask 8.1 GOM2 Sample Preparation for Micro-CT
Planned Finish: 1/17/19
Actual Finish: In Progress

In the previous quarter, we have conducted hydrate growth experiments in a sandy silt sediment sample from GOM2 using xenon gas and another in a coarse sand sediment using methane. During this quarter, we conducted a methane hydrate growth experiment in a loosely packed sandy silt GOM2 sediment sample (median grain size of 40 μm) at a resolution of 4.5 μm and find X-ray CT evidence of methane hydrate growth in pores as small as 20 μm. The sediment sample is from the core UT-GOM2-1-H005-06FB-2; depth of 429.46 - 429.56 m below sea floor and belongs to Lithofacies 2. Dissociation tests indicate that the sample originally had 84% hydrate saturation at in situ condition, and laser particle analysis measured 35% sand, 60% silt and 5% clay composition (data from UT-GOM2-1 Hydrate Pressure Coring Expedition Report - https://ig.utexas.edu/files/2018/02/1.0-UT-GOM2-1-Expedition-Summary.pdf).

Sample preparation consists of: 1) mixing the GOM2 sediment sample with 4.37 wt% KI brine before packing, 2) packing the damp sediments in a micro-consolidation device (3.6 mm-diameter and 10 mm-long), and 3) taking the sample into the hydrate stability zone under excess gas conditions. The damp-sand packing method results in large pores unlikely to be present in natural sediments. The initial step consists in increasing methane gas pressure to 8.14 MPa at room temperature (23.0°C). Then, we decrease the temperature to 2.7°C, within the hydrate stability zone. Figure 8.1 shows the experimental pressure-temperature path along with the CH_4 hydrate phase boundaries for pure water and 4.37 wt% brine.

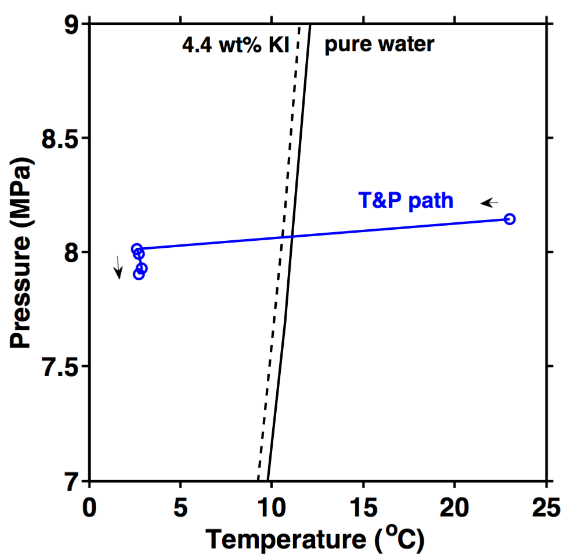

Figure 8.1. Experimental pressure-temperature path for methane hydrate formation in GOM2 Lithofacies 2 sample and CH_4 hydrate phase boundaries.

Figure 8.2 shows micro-CT slices of the sediment before and after 8 days into the hydrate stability zone. Figure 8.3 plots histograms of selected regions (large and small boxes in Figure 8.2).
Outside the stability zone: Methane gas occupies pores (black) with different sizes in the image outside the stability zone. For instance, the large white box (400 μm) shows a large pore about 400 μm by 60 μm, and the small white box (150 μm) shows pores sized about

20 μm. These large pores are a result of the damp-sand packing procedure. Smaller pores are saturated with brine. It is difficult to differentiate between brine and grains due to their similar CT numbers and the small grain size. The histograms of selected boxes show two distinct peaks for methane gas and the combination of sand and brine.

After 8 days within the hydrate stability zone: many of the previously gas-filled pores exhibit methane hydrate growth. For example, the CT numbers in the large pores within the selected boxes increase after 8 days within the stability zone. This change indicates the existence of methane hydrates within these large pores. Figure 8.3 plots the histograms that quantitatively demonstrate the presence of hydrate. After 8 days within the stability zone, the histogram of the large box shows that the gas peak decreased in size and there is only one major peak, due to the conversion of gas to hydrate (hydrate has higher CT number than gas). In addition, the histogram range for the small box shrinks by 6000 CT units after 8 days within the stability zone, a result of methane hydrate growth and gas consumption.

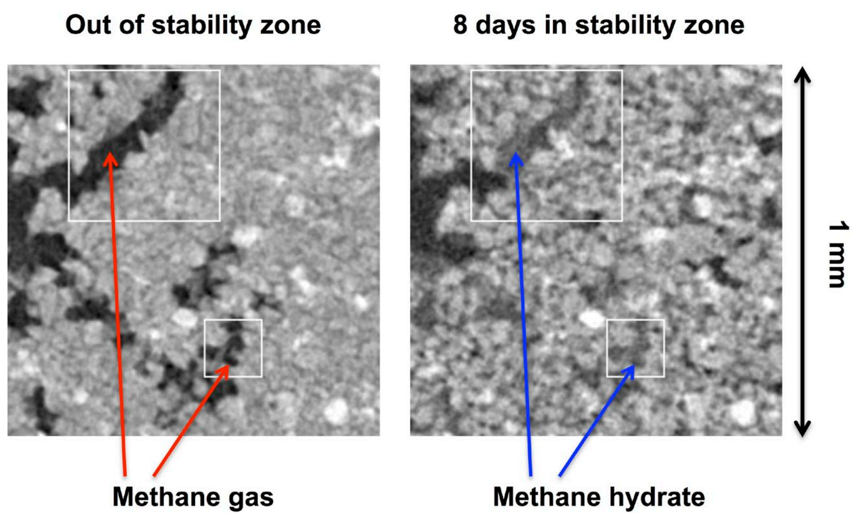

Figure 8.2. Micro-CT images of loosely packed GOM2 sediments out of the hydrate stability zone (left) and after 8 days within the CH_4 hydrate stability zone at a resolution of 4.5 μm. CT images identify hydrate only in large pores.

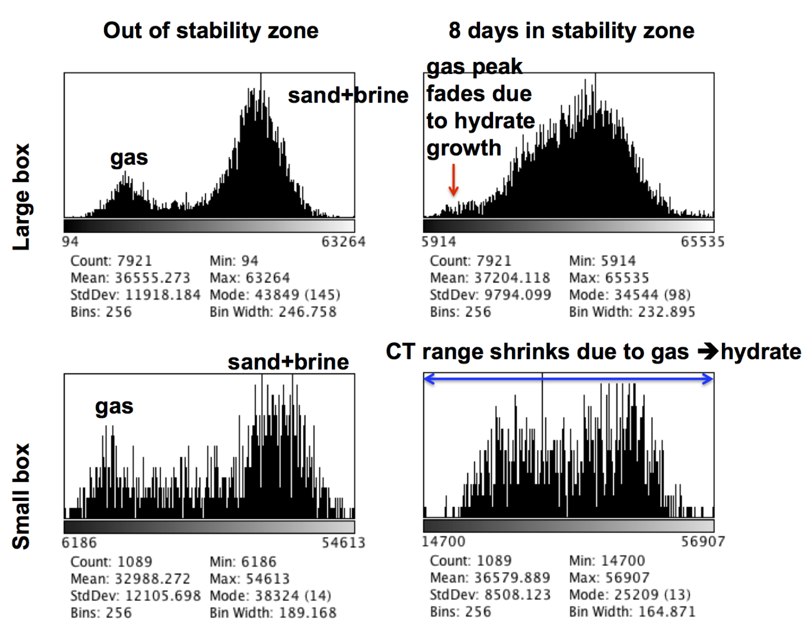

Figure 8.3. Histograms of selected regions in Figure 8.2.

Although we present evidence of hydrate formation in relatively large pores >20 µm. These pores are the result of the damp-sand packing method and unlikely to be present in natural sediments. The current apparatus and technique do not permit segmenting hydrate in the pore space of natural Lithofacies 2 sandy silt. We are working on alternatives to overcome these shortcomings.

Link to actions for next Quarter, Task 8

Subtask 8.2 Production Testing on GOM2 Samples Observed with Micro-CT
Planned Finish: 9/30/19
Actual Finish: In Progress

Methane hydrate dissociation experiment in coarse sediments can provide insights for understanding production test in GOM2 samples. We have conducted experiments within this quarter. Data processing is still undergoing and results will be provided in the next quarterly report.

Link to actions for next Quarter, Task 8

Task 9.0 Micro-Scale: Raman Observation on hydrate-bearing sand packs

Subtask 9.1 3D Imaging of methane hydrate sandpacks
Planned Finish: 1/17/19
Actual Finish: In Progress

In the previous quarter, we have been conducting a methane hydrate formation experiment (experiment number RH010). We loaded 2 kinds of sediments in our Raman chamber—sandy silt and clay-free quartz sand. The key difference in those 2 sediments is their grain size distributions. The sandy silt sample is from core GC955-H005-06FB-2 (Lithofacies 2) at a depth of 429.46 - 429.56 meter below sea floor. The natural sand is substantially coarser than sandy silt, with diameters ranging from 210 µm to 297 µm. We first loaded dry sediments in the chamber. A filter paper was inserted to separate two kinds of sediment and limit clay migrations from Lithofacies 2 to sand (Figure 9.1). We then loaded vapor methane and 3.5 wt% NaCl aqueous solution. The temperature was lowered into the hydrate stability zone to synthesize methane hydrate. Optically, hydrate formation is indicated by the disappearance of CH_4 vapor (Figure 9.2), accompanied by ~ 1 MPa drop in pressure.

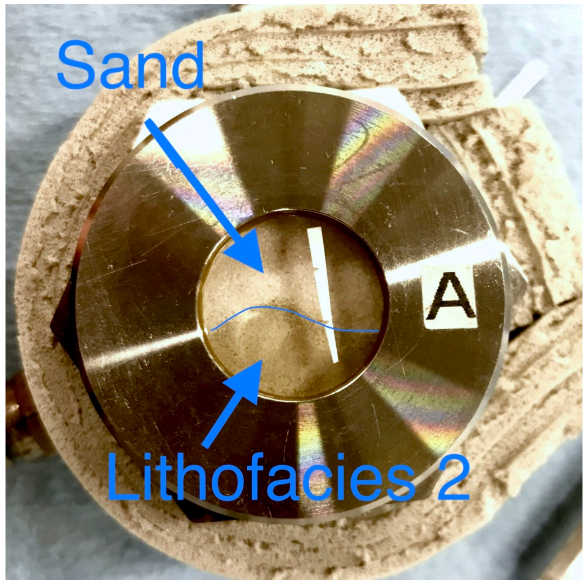

Figure 9.1. Photo of 2 kinds of dry sediments loaded in the Raman chamber prior to hydrate formation: sandy silt from core GC955-H005-06FB-2 (Lithofacies 2) and natural quartz sand. The mass medium diameter of Lithofacies 2 is 40 μm). The diameters of natural sand range from 210 μm to 297 μm.

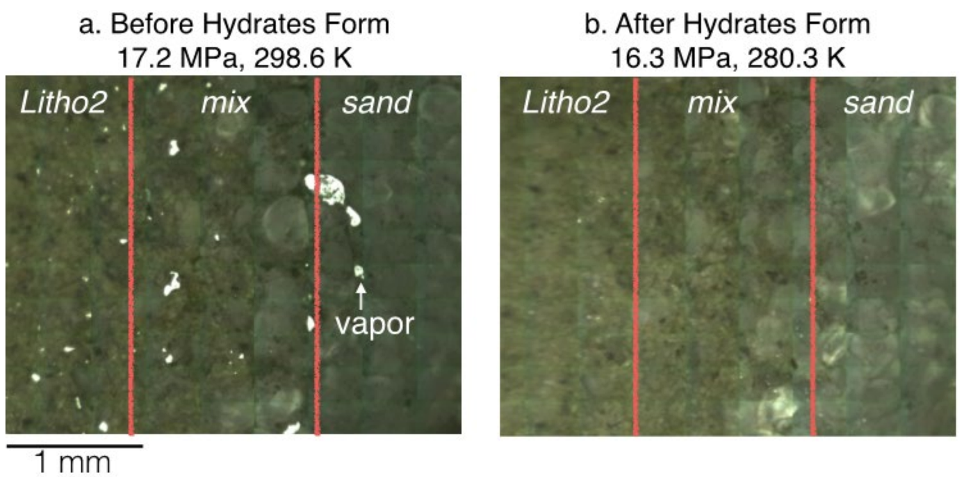

Figure 9.2. Optical images of the sample chamber before (a) and after (b) hydrate formation. The obvious signature of hydrate formation is the disappearance of vapor phase. The red vertical lines divide the sample chamber into three parts: Lithofacies 2 in the left; a mixture of Lithofacies 2 and natural sand in the middle and natural sand in the right.

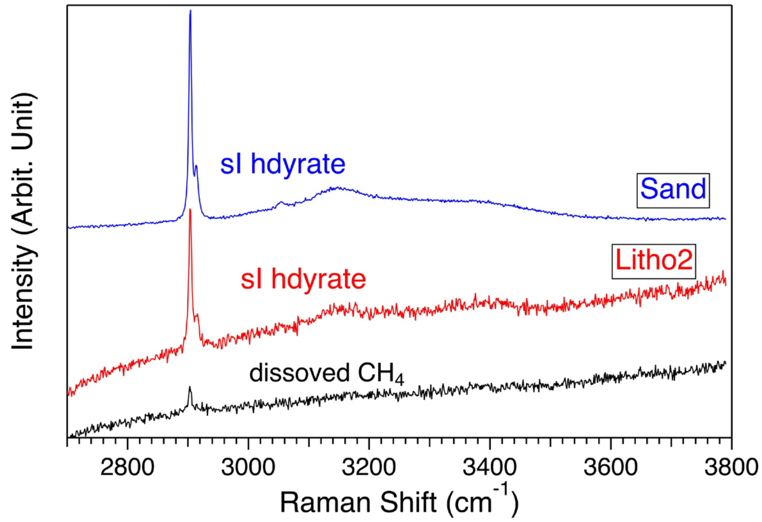

Figure 9.3. Representative Raman spectra of structure I (sI) methane hydrate in natural sand sample (blue) and Lithofacies 2 (red), together with dissolved methane vapor (black). The strong fluorescence from clay minerals make the background of the red spectrum tilted.

Link to actions for next Quarter, Task 9

Subtask 9.2 Micro-Raman Imaging of methane hydrate sandpacks
Planned Finish: 9/30/19
Actual Finish: In Progress

We deployed a Raman spectrometer to conduct 2D mapping over an area of 6500 µm by 2800 µm, which covers both Lithofacies 2, natural sand and their mixture in between (Figure 9.4). Each Raman data acquisition location is 50 µm apart in both X and Y directions. Thus, we acquired 7467 Raman spectra (131x 57) in each 2D mapping.

The time and spatial dependences of methane hydrates in Lithofacies 2 and natural sand are shown in Figure 9.4. During the initial stage in hydrate formation (Figure 9.4a and 9.4b), hydrates crystallized in all three sections of Lithofacies 2, mixture and natural sand. Compared with hydrates in natural sand, hydrates crystallizing in Lithofacies 2 had higher large cage/ small cage (LC/SC) ratios. Over time, less hydrates formed in Lithofacies 2 (Figure 9.4c and Figure 9.4e) than both sections of mixture and natural sand. This can be explained by the stronger capillary effect in Lithofacies 2 due to its smaller grain sizes than natural sand. Such a migration of hydrates also increased the LC/SC ratios of hydrates in the segments of mixture and natural sand, which are approaching the ideal LC/SC ratio of sI hydrate

We interpret that this evolution of hydrate concentration and position is driven by both nucleation behavior and capillary effects. Initially, methane hydrates formation was controlled by nucleation. As we lowered the temperature to form hydrate, a large number of nucleation sites were created simultaneously in both Lithofacies 2 and sand. Over time, the capillary effect is pronounced. Because the grains are smaller in Lithofacies 2 than in the clean sand, the solubility of the methane in water is higher. As a result, hydrates in the small pores dissolves and reforms in the large pores in the clean sand. The higher capillary force in Lithofacies 2 caused a dissolved methane concentration gradient from Lithofacies 2 to sand. Over time, methane migrated from Lithofacies 2 to sand through diffusion, lowering the methane hydrate concentration in Lithofacies 2 and elevating the hydrate concentration in natural sand (Figure 9.4a, c and e).

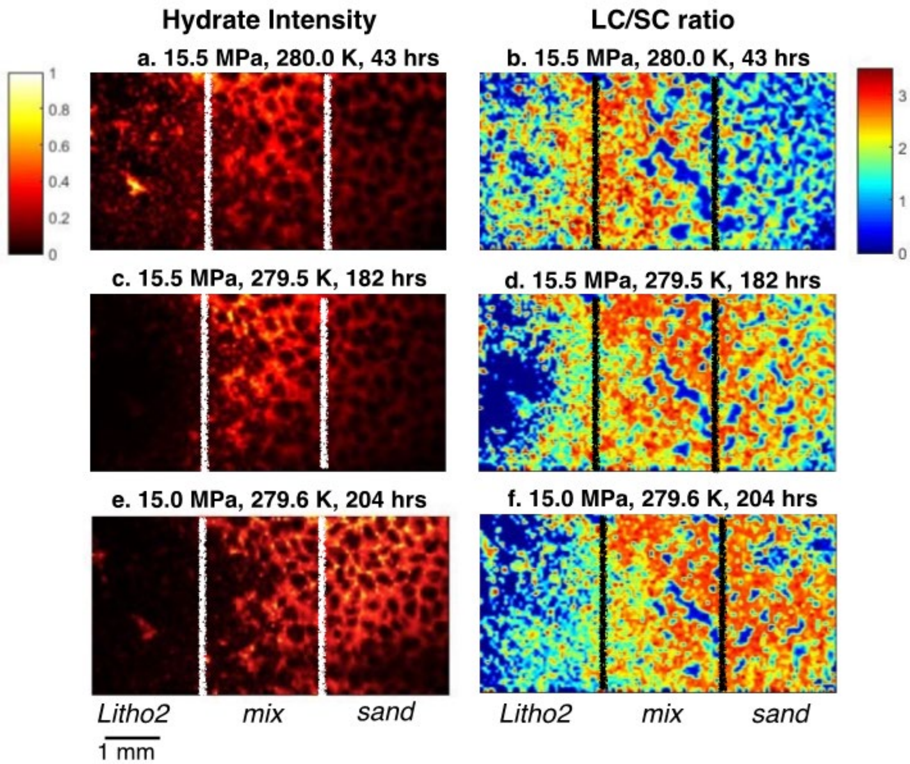

Figure 9.4. The spatial heterogeneity of hydrate contents and structures from 2-D Raman mappings. The two columns are Raman intensities of CH_4 peaks in hydrates and the corresponding large cage/ small cage (LC/SC) ratios. The three rows are data collected at different time. In each mapping, the lithofacies 2 sample is on the left and natural sand sample is on the right, while the middle part is a mixture of lithofacies 2 and natural sand samples.

C. What opportunities for training and professional development has the project provided?

We provided technical training and mentoring to 1 high school student and two early college-age students. These students participate in experimental design, research meetings, and experimental measurements. We continue to train 2 doctoral students and 3 post-doctoral scientists.

D. How have the results been disseminated to communities of interest?

- A presentation was made at the Third Deep Carbon Observatory International Science Meeting, St. Andrews, Scotland, 23-25, March.
- A poster was presented at the 9th International Conference on Gas Hydrates, June 25-30, 2017, Denver, CO.
- A poster was presented at the American Geophysical Union Fall Meeting 2017, Dec. 11-15, 2017, New Orleans, LA.
- An invited talk was given at the American Geophysical Union Fall Meeting 2017, December 11-15, 2017, New Orleans, LA.
- Two posters were presented at the Gordon Research Conference- Natural Gas Hydrate Systems, 2018, Feb 25 – March 2, Galveston, TX

- Steve Phillips presented an update on HP3 at the DOE Mastering the Subsurface Through Technology Innovation, Partnerships, and Collaboration: Carbon Storage and Oil and Natural Gas Technologies Review Meeting in August 2018 in Pittsburgh, PA.

E. What do you plan to do during the next reporting period to accomplish the goals?

Task 1.0 Project Management and Planning (next quarter plans)

Planned Finish: 09/30/19
Actual Finish: In progress

- Complete the Y2Q4 Quarterly
- Update the HP3 Website

Task 2.0 Macro-Scale: Relative Permeability of Methane Hydrate Sand Packs

Subtask 2.1 Laboratory Creation of Sand-Pack Samples at Varying Hydrate Levels
Planned Finish: 6/27/17
Actual Finish: 6/27/17

Subtask 2.2 Steady-State Permeability of Gas and Water of Sand-Pack Hydrate Samples
Planned Finish: 3/27/18
Actual Finish: 3/27/18

Task 3.0 Macro-Scale: Depressurization of Methane Hydrate Sand Packs

Subtask 3.1 Depressurization Tests
Planned Finish: 6/27/17
Actual Finish: 6/27/17

Subtask 3.2 Depressurization Tests with CAT scan
Planned Finish: 3/27/18
Actual Finish: 3/27/18

Task 4.0 Micro-Scale: CT Observation of Methane Hydrate Sand Packs

Subtask 4.1 Design and Build a Micro-CT compatible Pressure Vessel
Planned Finish: 6/27/17
Actual Finish: 6/27/17

Subtask 4.2 Micro-Scale CT Observations and Analysis
Planned Finish: 3/27/18
Actual Finish: 3/27/2018

Task 5.0 Micro-Scale: Raman Observation of Methane-Gas-Water Systems

Subtask 5.1 Design and Build a Micro-Raman compatible Pressure Vessel
Planned Finish: 6/27/17
Actual Finish: 6/27/17

Subtask 5.2 Micro-scale petrochemistry
Planned Finish: 03/21/18
Actual Finish: 3/27/18

Subtask 5.2 Diffusion kinetics of methane release
Planned Finish: 03/27/18
Actual Finish: 3/27/18

Task 6.0 Macro-Scale: Relative Permeability of Methane Hydrate Sand Packs and Intact Pressure Core Samples (next quarter plans)

Subtask 6.1 Steady-State Relative Permeability Measurements of Sand-Pack Hydrate Samples
Planned Finish: 1/17/19
Actual Finish: In Progress

We will now run our experiments in a cooling jacket which will allow more precise control of the temperature conditions as well as the ability to CT scan the core. We will be able to scan the core before hydrate formation to determine porosity and water saturation/distribution, after hydrate formation to determine hydrate saturation and distribution, during injection to determine changing phase saturations, and after injection to determine any changes to hydrate saturation. This improvement will allow us to constrain our relative permeability values to phase saturations and have a better understanding of our initial and final hydrate distribution. In order to measure each phase saturation within the core, we plan to CT scan the core during three-phase permeability experiments to measure phase saturations. To do this, we designed a cooling jacket, which will allow us to maintain the temperature of the core at 6 °C during scanning. CT images will give a more accurate assessment of each phase (hydrate, water, and gas) saturation as well as a better understanding of how hydrate moves throughout our experiments. This cooling jacket is currently being built and should be completed soon.

We previously had been performing our experiments at pore pressures of 1200 psi, which required a brine salinity of 9.8% to maintain three-phase stability. The high salinity has been damaging our equipment, so we have decided to conduct our future experiments at a pore pressure of 770 psi. This lower pressure will only require a brine salinity of 2.9%.

We also decided to use Berea sandstone cores with permeabilities of 100-500 md rather than sand packs or Boise sandstone cores. There are two reasons for this change. First, the absolute permeabilities of Berea cores are more representative of Gulf of Mexico sediments. Furthermore, changing flow and saturation conditions in sand packs can alter the grain arrangement, which will subsequently alter the absolute permeability. Measuring relative permeability in the absence of changing geomechanical properties is important, and since sand pack properties are highly dependent on the applied pressures, by using sandstone cores we can decouple flow properties from geomechanical effects.

Finally, we will address the issue of the differential pressure transducers not measuring a value of 0 when no flow is occurring. This may be due to hydrate formation or redistribution continuing even after gas injection stops. CT scans may help diagnose this. Running experiments with a lower salinity fluid may also help alleviate some issues involving corrosion, abrasion, and salt precipitation, which may contribute to the issue.

Subtask 6.2 Steady-State Relative Permeability Measurements of Intact Pressure Cores
Planned Finish: 9/30/19
Actual Finish:

We will start this task by 5/1/19

Task 7.0 Macro-Scale: Depressurization of Methane Hydrate Sand Packs and Intact Pressure Core Samples (next quarter plans)

Subtask 7.1 Depressurization of sand-pack hydrate samples
Planned Finish: 1/17/19
Actual Finish: In Progress

We will form hydrates using the formation method used in Task 2.0 to obtain hydrate saturations > 40% and then depressurize while observing pressure rebound behavior. This will allow us to observe the influence of hydrate saturation and formation method on the form of pressure rebounds.

Subtask 7.2 Depressurization of intact pressure cores
Planned Finish: 9/30/19
Actual Finish: In Progress

We will continue depressurize pressure core samples recovered during the UT-GOM2-1 Expedition. We will slowly depressurize hydrate-bearing samples of sandy silt and clayey silt while monitoring pressure rebounds between steps during dissociation. This approach will allow us to observe the influence of lithology and hydrate saturation on pressure recovery behavior during dissociation. We will look at the influence of lithofacies (sandy silt vs. clayey silt) and hydrate saturation (5 to 93%) on pressure rebound behavior.

Task 8.0 Micro-Scale: CT experiments on Gulf of Mexico Sand Packs (next quarter plans)

Subtask 8.1 GOM2 Sample Preparation for Micro-CT
Planned Finish: 1/17/19
Actual Finish: In Progress

We plan to improve the packing method for the GOM2 Lithofacies 2 samples and redo CH_4 hydrate formation experiments. We will use volume average “segmentation-less” methods to prove the existence of hydrate formation using our facilities at a maximum resolution of 4.5 μm.
The current image resolution (pixel size 4.5 μm) does not enable reliable segmentation in sandy-silt grain packs. We are evaluating the use of alternative X-ray scanning devices with higher resolution (<1 μm). Such facilities are available at UT Austin and NETL. We will contact Y. Seol and K. Jarvis at NETL to investigate possibilities for collaboration.
We will take a closer look at GOM2 cores to investigate if there are coarser sediment sections that could be used for micro-CT hydrate experiments.

Subtask 8.2 Production Testing on GOM2 Samples Observed with Micro-CT
Planned Finish: 9/30/19
Actual Finish: In Progress

- We will provide results of CH_4 hydrate dissociation in coarse sands.
- We will dissociate the current methane hydrate experiment in GOM2 sample and report the results

Task 9.0 Micro-Scale: Raman Observation on hydrate-bearing sand packs (next quarter plans)

Subtask 9.1 3D Imaging of methane hydrate sandpacks
Planned Finish: 1/17/19
Actual Finish: In Progress

- We will dissociate the ongoing methane hydrate experiment in sand and Lithofacies 2.

Subtask 9.2 Micro-Raman Imaging of methane hydrate sandpacks
Planned Finish: 9/30/19
Actual Finish: In Progress

- We will repeat a previous experiment with silica glass beads in an attempt to reproduce the previous results and collect additional data.
- We will assemble another experiment with Lithofacies 2 and Lithofacies 3 loaded, with similar configuration as experiment RH010 (Fig. 9.1). This experiment will enable us to understand how hydrates crystallize and migrate in GOM^2 pressure-temperature-composition conditions.

2. PRODUCTS:

What has the project produced?

a. Publications, conference papers, and presentations

Dong, T., Lin, J. F., Flemings, P. B., Polito, P. J. (2016), Pore-scale study on methane hydrate dissociation in brine using micro-Raman spectroscopy, presented at the 2016 Extreme Physics and Chemistry workshop, Deep Carbon Observatory, Palo Alto, Calif., 10-11 Dec.

Lin, J. F., Dong, T., Flemings, P. B., Polito, P. J. (2017), Characterization of methane hydrate reservoirs in the Gulf of Mexico, presented at the Third Deep Carbon Observatory International Science Meeting, St. Andrews, Scotland, 23-25, March.

Phillips, S.C., You, K., Flemings, P.B., Meyer, D.W., and Dong, T., 2017. Dissociation of laboratory-synthesized methane hydrate in coarse-grained sediments by slow depressurization. Poster presented at the 9th International Conference on Gas Hydrates, June 25-30, 2017, Denver, CO.

Chen, X., Espinoza, N., Verma, R., and Prodanovic, M. X-Ray Micro-CT Observations of Hydrate Pore Habit and Lattice Boltzmann Simulations on Permeability Evolution in Hydrate Bearing Sediments (HBS). Presented at the 2017 AGU Fall Meeting, December 11-15, 2017, New Orleans, LA.

Chen, X., & Espinoza, D. N. (2018). Ostwald ripening changes the pore habit and spatial variability of clathrate hydrate. Fuel, 214, 614–622. https://doi.org/10.1016/j.fuel.2017.11.065

Chen, X., Verma, R., Nicolas Espinoza, D., & Prodanović, M. (2018). Pore-Scale Determination of Gas Relative Permeability in Hydrate-Bearing Sediments Using X-Ray Computed Micro-Tomography and Lattice Boltzmann Method. Water Resources Research, 54(1), 600-608. https://doi.org/10.1002/2017WR021851

Chen, X and Espinoza, DN (2018), Surface area controls gas hydrate dissociation kinetics in porous media, Fuel, 234, 358-363. https://doi.org/10.1016/j.fuel.2018.07.030

Xiongyu Chen, D. Nicolas Espinoza, Nicola Tisato, Peter B. Flemings (2018). X-ray Computed Micro-Tomography Study of Methane Hydrate Bearing Sand: Enhancing Contrast for Improved Segmentation, Gordon Research Conference – Natural Gas Hydrate Systems, Galveston, TX

Xiongyu Chen, D. Nicolas Espinoza, Nicola Tisato, Rahul Verma, Masa Prodanovic, Peter B. Flemings, (2018). New Insights Into Pore Habit of Gas Hydrate in Sandy Sediments: Impact on Petrophysical and Transport Properties, Gordon Research Conference – Natural Gas Hydrate Systems, Galveston, TX

Dong, T., Lin, J.-F., Flemings, P.B., Gu, J.T., Liu, J., Polito, P.J., O'Connell, J. (2017) Pore-scale study on gas hydrate formation and dissociation under relevant reservoir conditions of the Gulf of Mexico, presented at the 2017 Extreme Physics and Chemistry workshop, Deep Carbon Observatory, November 4-5, Tempe, AZ.

Dong, T., Lin, J.-F., Gu, J.T., Polito, P.J., O'Connell, J., Flemings, P.B. (2017), Spatial and temporal dependencies of structure II to structure I methane hydrate transformation in porous media under moderate pressure and temperature conditions, Abstract OS53B-1188 Presented at 2017 Fall Meeting, December 11-15, New Orleans, LA.

Dong, T., Lin, J.-F., Gu, J.T., Polito, P.J., O'Connell, J., Flemings, P.B. (2018), Transformation of metastable structure-II to stable structure-I methane hydrate in porous media during hydrate formation, poster presented at 2018 Jackson School of Geosciences Symposium, Feb. 3, 2018, Austin, TX.

Dong, T., Lin, J.-F., Flemings, P.B., Gu, J.T., Polito, P.J., O'Connell, J. (2018), Pore-scale methane hydrate dissociation in porous media using Raman spectroscopy and optical imaging, poster presented at Gordon Research Conferences on Natural Gas Hydrate Systems, Feb. 25-March 2, 2018, Galveston, TX.

Meyer, D.W., Flemings, P.B., DiCarlo, D., You, K., Phillips, S.C., and Kneafsey, T.J. (2018), Experimental investigation of gas flow and hydrate formation within the hydrate stability zone. Journal of Geophysical Research- Solid Earth https://doi.org/10.1029/2018JB015748

Meyer, D., Flemings, P.B., DiCarlo, D. (submitted), Effect of Gas Flow Rate on Hydrate Formation Within the Hydrate Stability Zone, Journal of geophysical research

Meyer, D., PhD Dissertation (submitted) Dynamics of Gas Flow and Hydrate Formation within the Hydrate Stability Zone

Murphy, Z., Fukuyama, D., Daigle, H., DiCarlo, D. (2018), Relative permeability of hydrate-bearing sediment, poster presented at Gordon Research Conference on Natural Gas Hydrate Systems, Feb. 25-Mar. 2, 2018, Galveston, TX.

b. Website(s) or other Internet site(s)

- Project SharePoint: https://sps.austin.utexas.edu/sites/GEOMech/HP3/_layouts/15/start.aspx#/SitePages/Home.aspx
- Project Website

https://ig.utexas.edu/energy/hydrate-production-properties/

c. Technologies or techniques

Nothing to Report.

d. Inventions, patent applications, and/or licenses

Nothing to Report.

e. Other products

Research Performance Progress Report (Period ending 12/31/16)
Research Performance Progress Report (Period ending 3/31/17)
Research Performance Progress Report (Period ending 6/30/17)
Research Performance Progress Report (Period ending 9/30/17)
Research Performance Progress Report (Period ending 12/31/17)
Research Performance Progress Report (Period ending 3/31/18)
Phase 1 Report (Period ending 3/31/18)
Research Performance Progress Report (Period ending 6/30/18)

3. CHANGES/PROBLEMS:

This section highlights changes and problems encountered on the project.

a. Changes in approach and reasons for change

- Relative Permeability Experiments (Task 6): We had significant challenges developing consistent pressure drops in our sections in our relative permeability experiments using sand packs. We ultimately changed from performing these experiments on sand-packs to performing these experiments on Boise sandstone core. We are now making successful relative permeability measurements on the sandstone core. We may return to examining relative permeability in sand packs after completion of analysis of relative permeability on the sandstone core. To improve our ability to measure relative permeability with reasonable pressure drops, we have opted to move forward with Berea sandstone, which has lower intrinsic permeability than Boise sandstone but still within the range expected for Gulf of Mexico hydrate reservoirs. A second major challenge is our ability to determine water and gas saturations during the measurements. The phase saturations are not simply a scaled function of the relative injection ratios due to the presence of residual water saturation. Related to this is the apparent heterogeneity of hydrate distribution evident in the relative permeability measurements (Figures 2-5). We plan to use CT scanning to help determine (a) the initial water saturation and hydrate distribution, and (b) gas saturation. The CT scans will be combined with weighing the core to help determine phase saturations as well as heterogeneity of distribution.

- Microscale Imaging (Task 8): It has been challenging to develop sufficient contrast to image gas, methane hydrate, and brine. For this reason, we have changed brines, decreased the sample diameter and increased the imaging resolution. We will be using potassium iodide (KI) salts for the ensuing experiments and extend experiments to 1/8 in diameter.

b. Actual or anticipated problems or delays and actions or plans to resolve them

Nothing to Report.

c. Changes that have a significant impact on expenditures

Nothing to Report.

d. Change of primary performance site location from that originally proposed

Nothing to Report.

4. SPECIAL REPORTING REQUIREMENTS:

Special reporting requirements are listed below.

PAST - BUDGET PERIOD 1

Nothing to Report

CURRENT – BUDGET PERIOD 2

Nothing to Report.

5. BUDGETARY INFORMATION:

The Cost Summary is located in Exhibit 1.

EXHIBIT 1 – COST SUMMARY

Baseline Reporting Quarter	Budget Period 1 (Year 1)							
	Q1		Q2		Q3		Q4	
	10/01/16-12/31/16		01/01/17-03/31/17		04/01/17-06/30/17		07/01/17-09/30/17	
	Q1	Cumulative Total	Q2	Cumulative Total	Q3	Cumulative Total	Q4	Cumulative Total
Baseline Cost Plan								
Federal Share	$ 283,497	$ 283,497	$ 82,038	$ 365,535	$ 79,691	$ 445,226	$ 79,691	$ 524,917
Non-Federal Share	$ 170,463	$ 170,463	$ 7,129	$ 177,593	$ 7,129	$ 184,722	$ 7,129	$ 191,851
Total Planned	$ 453,960	$ 453,960	$ 89,167	$ 543,128	$ 86,820	$ 629,948	$ 86,820	$ 716,768
Actual Incurred Cost								
Federal Share	$ 6,749	$ 6,749	$ 50,903	$ 57,652	$ 67,795	$ 125,447	$ 162,531	$ 287,977
Non-Federal Share	$ 10,800	$ 10,800	$ 10,800	$ 21,600	$ 10,800	$ 32,400	$ 158,478	$ 190,878
Total Incurred Cost	$ 17,549	$ 17,549	$ 61,703	$ 79,252	$ 78,595	$ 157,847	$ 321,009	$ 478,855
Variance								
Federal Share	$ (276,748)	$ (276,748)	$ (31,135)	$ (307,883)	$ (11,896)	$ (319,779)	$ 82,840	$ (236,940)
Non-Federal Share	$ (159,663)	$ (159,663)	$ 3,671	$ (155,993)	$ 3,671	$ (152,322)	$ 151,349	$ (973)
Total Variance	$ (436,411)	$ (436,411)	$ (27,465)	$ (463,876)	$ (8,226)	$ (472,101)	$ 234,188	$ (237,913)

Baseline Reporting Quarter	Budget Period 1 & 2 (Year 2)							
	Q1		Q2		Q3		Q4	
	10/01/17-12/31/17		01/01/18-03/31/18		04/01/18-06/30/18		07/01/18-09/30/18	
	Q1	Cumulative Total	Q2	Cumulative Total	Q3	Cumulative Total	Q4	Cumulative Total
Baseline Cost Plan								
Federal Share	$ 109,248	$ 634,165	$ 89,736	$ 723,901	$ 128,914	$ 852,815	$ 106,048	$ 958,863
Non-Federal Share	$ 7,342	$ 199,193	$ 19,369	$ 218,562	$ 7,342	$ 225,904	$ 31,393	$ 257,297
Total Planned	$ 116,590	$ 833,358	$ 109,105	$ 942,463	$ 136,256	$ 1,078,719	$ 137,441	$ 1,216,160
Actual Incurred Cost								
Federal Share	$ 107,216	$ 395,193	$ 154,758	$ 549,951	$ 163,509	$ 713,460	$ 161,083	$ 874,542
Non-Federal Share	$ 19,857	$ 210,735	$ 7,140	$ 217,875	$ 32,567	$ 250,442	$ 7,241	$ 257,683
Total Incurred Cost	$ 127,073	$ 605,928	$ 161,898	$ 767,826	$ 196,076	$ 963,902	$ 168,324	$ 1,132,225
Variance								
Federal Share	$ (2,032)	$ (238,972)	$ 65,022	$ (173,950)	$ 34,595	$ (139,355)	$ 55,035	$ (84,321)
Non-Federal Share	$ 12,515	$ 11,542	$ (12,229)	$ (687)	$ 25,225	$ 24,538	$ (24,152)	$ 386
Total Variance	$ 10,483	$ (227,430)	$ 52,793	$ (174,637)	$ 59,820	$ (114,817)	$ 30,883	$ (83,934)

Baseline Reporting Quarter	Budget Period 2 (Year 3)							
	Q1		Q2		Q3		Q4	
	10/01/18-12/31/18		01/01/19-03/31/19		04/01/19-06/30/19		07/01/19-09/30/19	
	Q1	Cumulative Total	Q2	Cumulative Total	Q3	Cumulative Total	Q4	Cumulative Total
Baseline Cost Plan								
Federal Share	$ 80,035	$ 1,038,898	$ 53,698	$ 1,092,596	$ 53,698	$ 1,146,294	$ 53,695	$ 1,199,989
Non-Federal Share	$ 7,581	$ 264,878	$ 7,579	$ 272,457	$ 7,579	$ 280,036	$ 19,965	$ 300,001
Total Planned	$ 87,616	$ 1,303,776	$ 61,277	$ 1,365,053	$ 61,277	$ 1,426,330	$ 73,660	$ 1,499,990
Actual Incurred Cost								
Federal Share								
Non-Federal Share								
Total Incurred Cost								
Variance								
Federal Share								
Non-Federal Share								
Total Variance								

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