



DOE Award No.: DE-FE-0028967

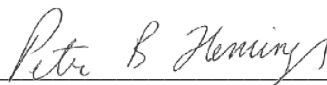
Quarterly Research Performance Progress Report (Period Ending 3/31/2018)

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

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

Submitted by:

Peter B. Flemings

A handwritten signature in cursive script, reading 'Peter B. Flemings', is positioned above a horizontal line.

Signature

The University of Texas at Austin

DUNS #: 170230239

101 East 27th Street, Suite 4.300

Austin, TX 78712-1500

Email: pflemings@jsg.utexas.edu

Phone number: (512) 475-8738

Prepared for:

United States Department of Energy

National Energy Technology Laboratory

April 30, 2018



U.S. DEPARTMENT OF
ENERGY

**NATIONAL ENERGY
TECHNOLOGY LABORATORY**

Office of Fossil Energy

DISCLAIMER

“This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.”

1. ACCOMPLISHMENTS:

What was done? What was learned?

This report outlines the progress of the second quarter of the second year of the first budget period. The majority of the progress made was completing initial experiments to meet Phase 1 Milestones.

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 will first demonstrate our ability to systematically manufacture sand-pack hydrate samples at a range of hydrate saturations. We will then 1) measure the permeability of the hydrate-saturated sand pack to flow of a single phase (water or gas), 2) depressurize the hydrate-saturated sand packs and observe the kinetic (time-dependent) behavior. Simultaneously we will build a micro-CT pressure container and a micro-Raman Spectroscopy chamber to image the pore-scale habit, phases, and pore fluid chemistry of our sand-pack hydrate samples. We will then make these 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)	11/22/2016	Presentation	Complete
Milestone 1.B: Achieve hydrate formation in sand-pack (See Subtask 2.1) Task 2.0 Macro-Scale:	6/27/2017 (Y1Q3)	8/11/2017	Documentation of milestone achievement within required project reporting / deliverables (Deliverable 2.1)	Complete, <i>Documentation in the Y1Q3 quarterly, and the upcoming Phase 1 report</i>
Milestone 1.C: Controlled and measured hydrate saturation using different methods (See Subtask 2.2) 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, <i>Documentation in this quarterly, and the upcoming Phase 1 report</i>
3 Milestone 1.D: Achieved depressurization and demonstrated mass balance (See Subtask 3.1) 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, <i>Documentation in the Y2Q1 Quarterly and upcoming Phase 1 report</i>
Milestone 1.E: Built and tested micro-consolidation device (See Subtask 4.1) 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, <i>Documentation in Y1Q3 Quarterly, Milestone report and upcoming Phase 1 report</i>
Milestone 1.F: Achieved Hydrate formation and measurements in Micro-CT consolidation device (See Subtask 4.2) 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, <i>Documentation in this quarterly, and the upcoming Phase 1 report</i>
Milestone 1.G: Built and integrated high-pressure gas mixing chamber (See Subtask 5.1) Task 5.0 Micro-Scale:	3/27/2018 (Y2Q2)	6/27/17	Documentation of milestone achievement within required project reporting / deliverables (Deliverable 5.1)	Complete, <i>Documentation in Y1Q3 Quarterly, and upcoming Phase 1 report</i>
Milestone 1.H: Micro-Raman analysis of synthetic complex methane hydrate (See Subtask 5.2 and 5.3) 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, <i>Documentation in this quarterly, and the upcoming Phase 1 report</i>
Milestone 2.A - Measurement of relative permeability in sand-pack cores.	1/17/2019 (Y3Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 6.1)	

Milestone 2.B - Measurement of relative permeability in intact pressure cores.	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.	9/30/2019 (Y3Q4)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 7.1)	
Milestone 2.D - Achieved gas production from GOM ² samples monitored by micro-CT.	9/30/2019 (Y3Q4)		Documentation of milestone achievement within required project reporting / deliverables Report (Deliverable 8.1)	
Milestone 2.E - Building a chamber to prepare natural samples for 2D-3D micro-Raman analysis;	1/17/2019 (Y3Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 9.1)	
Milestone 2.F - 2D micro-Raman analysis of natural methane hydrate samples at depressurization;	9/30/2019 (Y3Q4)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 9.1)	

B. What was accomplished under these goals?

CURRENT- BUDGET PERIOD 1

Task 1.0 Project Management and Planning

Planned Finish: 09/30/19

Actual Finish: In progress

The fifth Quarter Report was submitted on February 14, 2018. Request for Continuation (Continuation application) submitted on March 5, 2018.

[Link to Phase 2, Task 1, continued work](#)

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. Documentation of Milestone 1.B will be included in 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: In progress

After completing the construction of the system to conduct steady state relative permeability measurements, we have been testing the equipment with rock cores and are now conducting permeability measurements on hydrate bearing sediment. Due to the complications of sand packs and hydrate, we tested the system with a Berea Sandstone core to confirm that our setup worked with minimal fluid leakage. We conducted intrinsic permeability and relative permeability to gas measurements on the Berea core. The intrinsic permeability was 325 mD, and the relative permeability curve is shown in Figure 2.1. We decided that the results were acceptable and moved forward with tests with the sandpack.

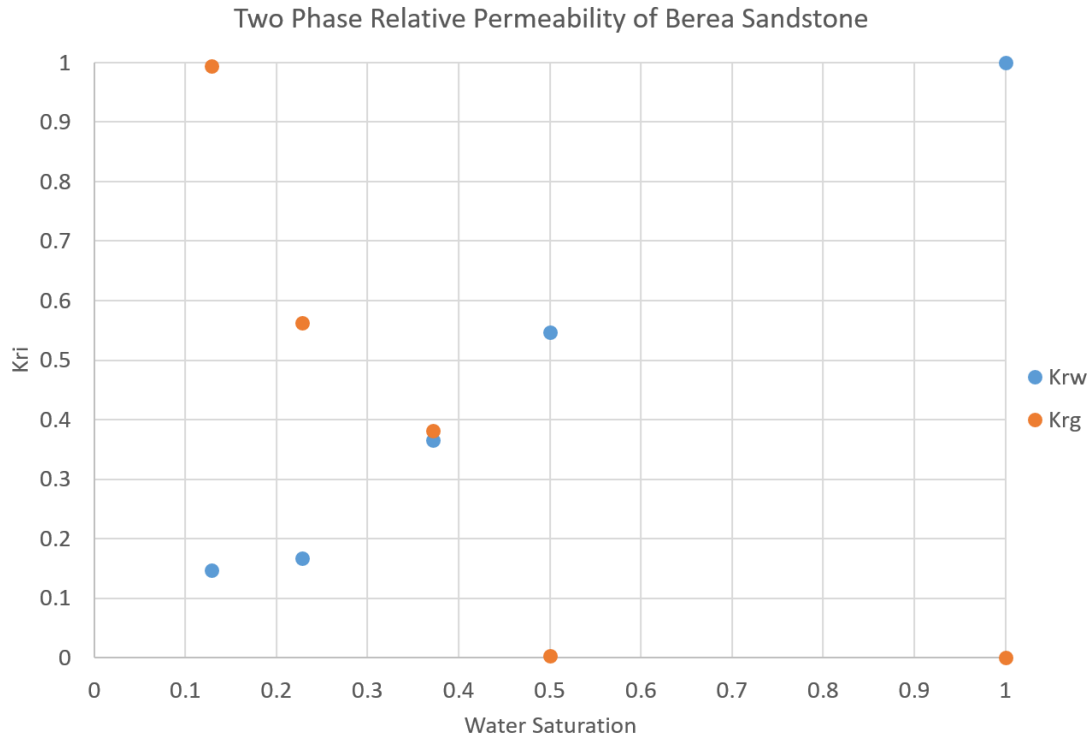


Figure 2.1. Relative permeability to water (blue) in gas (orange) in Berea sandstone measured with our experimental setup.

After dealing with some complications from pressurizing the sandpack, we began forming hydrates in the core holder with six pressure taps hooked up to the differential pressure transducers, as seen in Figure 2.2 below.

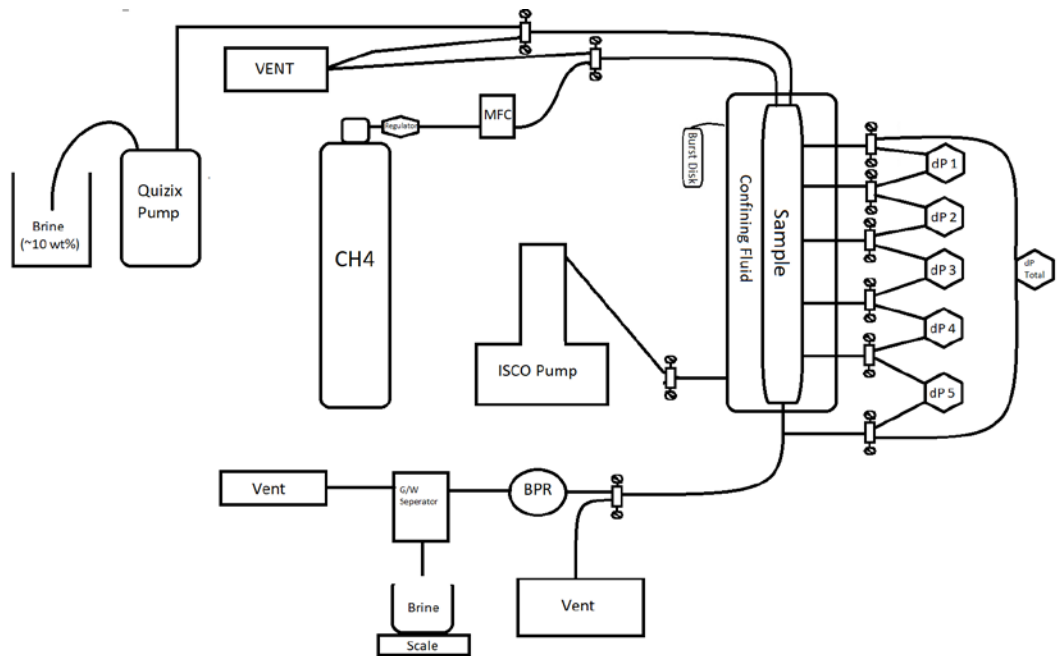


Figure 2.2. Experimental design.

The first step in our experiment is to pack the sample into the core holder with a known water saturation (~35%). Once the sample is packed, the confining pressure is increased to ~500 psi and a constant effective stress of 500 psi is applied to the sample. We then increase the pore pressure of the sample as we increase the confining pressure. Once the sample reaches 1250 psi and the confining is 1750 psi, we allow the sample to reach equilibrium. The pressure transducer lines are then opened to the core and pressure drops across the core are measured. At this point, since there is no flow, all pressure transducers should be reading 0 psi. After we ensure there are no leaks in the system, the entire setup/cart is transported in the cold room and allowed to reach experimental conditions ~-6°C. The pore pressure is controlled by an additional ISCO pump which is set to constant pressure mode at 1250 psi. As the system cools, gas is injected to maintain the pressure. After 6-15 hours, hydrate formation will begin and can be seen by the amount of gas injected by the pump. In order to allow maximum conversion, we allow the system to continue to form hydrate for 2-3 days.

Hydrate formation began after approximately 16 hours (Figure 2.3), and continued for more than 65 hours. Once hydrate formation is complete, the hydrate saturation is ~30% with a water conversion rate of 75%.

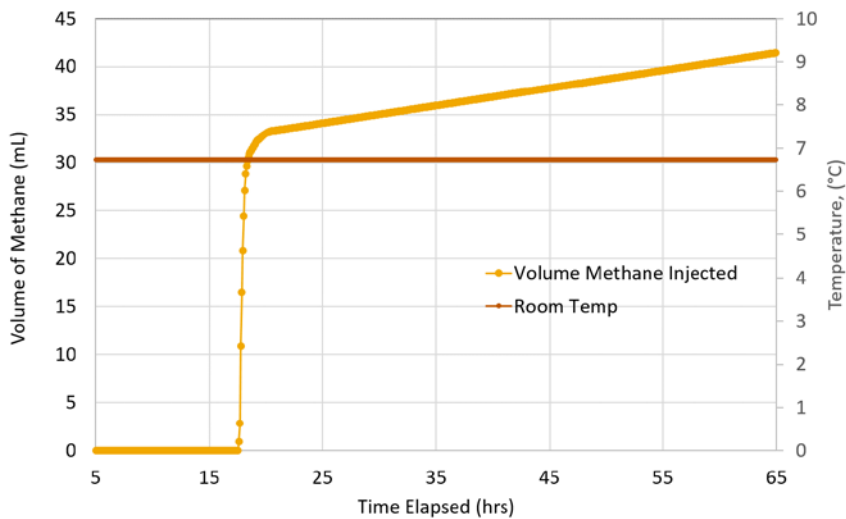


Figure 2.3. Hydrate formation using excess gas method. Methane pressure is 1250 psi.

The next step is to begin flowing three phase brine through the core. Since brine with buffer the formation of hydrates, we have calculated the salinity for three phase brine at our PT conditions (1250 psi and 6°C). At our conditions, the three phase brine is 10.5 wt% NaCl. We inject brine and bleed off any excess gas in the system until the sample is fully brine saturated. Once fully saturated, multiple flow rates are injected to determine the pressure drops and measure the effective permeability of the sample.

Challenges

We have currently created hydrate in our sandpack and are flowing brine through the sample. However, we are noticing that hydrate is forming/dissociating near/in the pressure taps which is blocking the pressure transducer lines. In Figure 2.4, hydrate is forming in/near the pressure tap shared by dP 4 and dP 5 causing extreme fluctuations in the pressure drops. Our theory is that hydrate is forming, causing the spikes in pressure, and then dissociating causing the dP to stabilize. This trend continues over 60+ hours of flow.

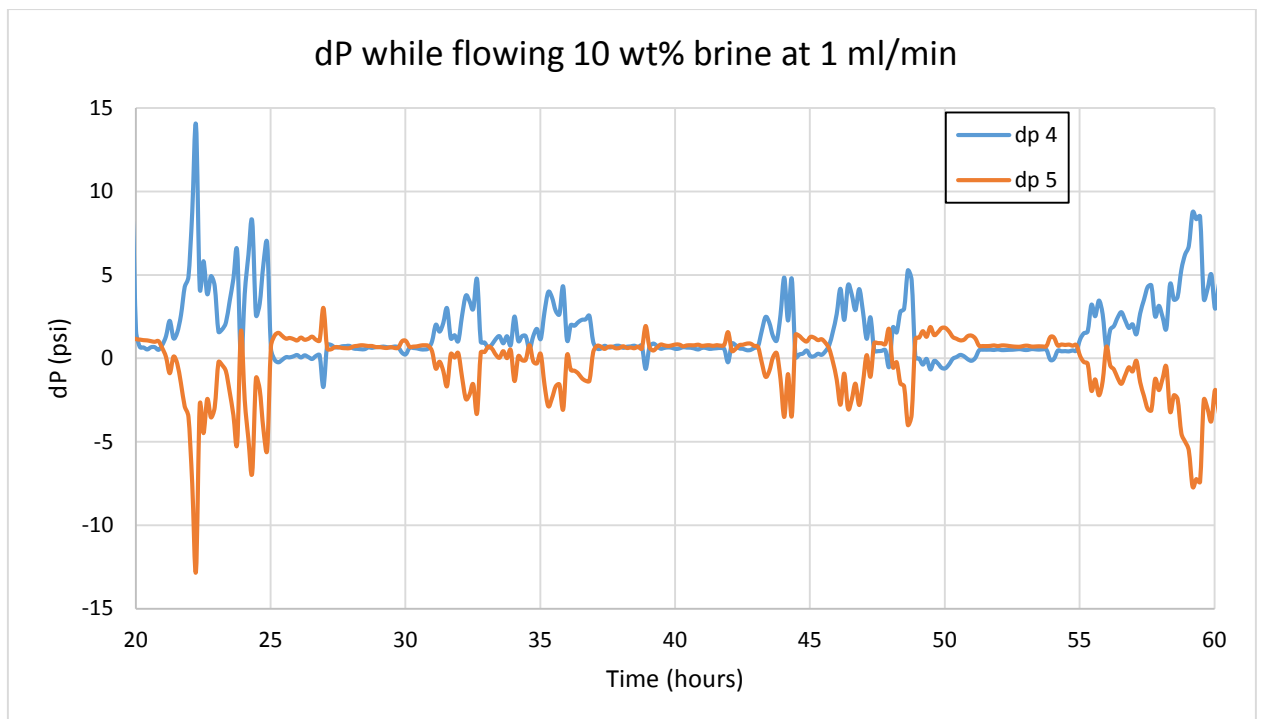


Figure 2.4. Pressure drop at 1 mL/min indicating hydrate blockage.

This blockage, which occurred at all pressure taps, prevents us from being able to accurately measure the pressure drop across the core. Additionally, since each tap is shared by two transducers, if one tap is blocked, two transducers are therefore ineffective. In order to solve this problem, we have filled the transducer lines with brine that is more saline than three phase stability (~13 wt% NaCl). This extremely saline brine will prevent hydrate formation when the brine comes into contact with methane gas. We have filled all transducer lines with this brine and have successfully prevented hydrate from forming in the lines (Figure 2.5). The difference in dP between the two pressure taps shown in Figure 2.5 is due to heterogeneity in the distribution of hydrate. However, we do still have a partial blockage of one pressure tap (not shown in Figure 2.5).

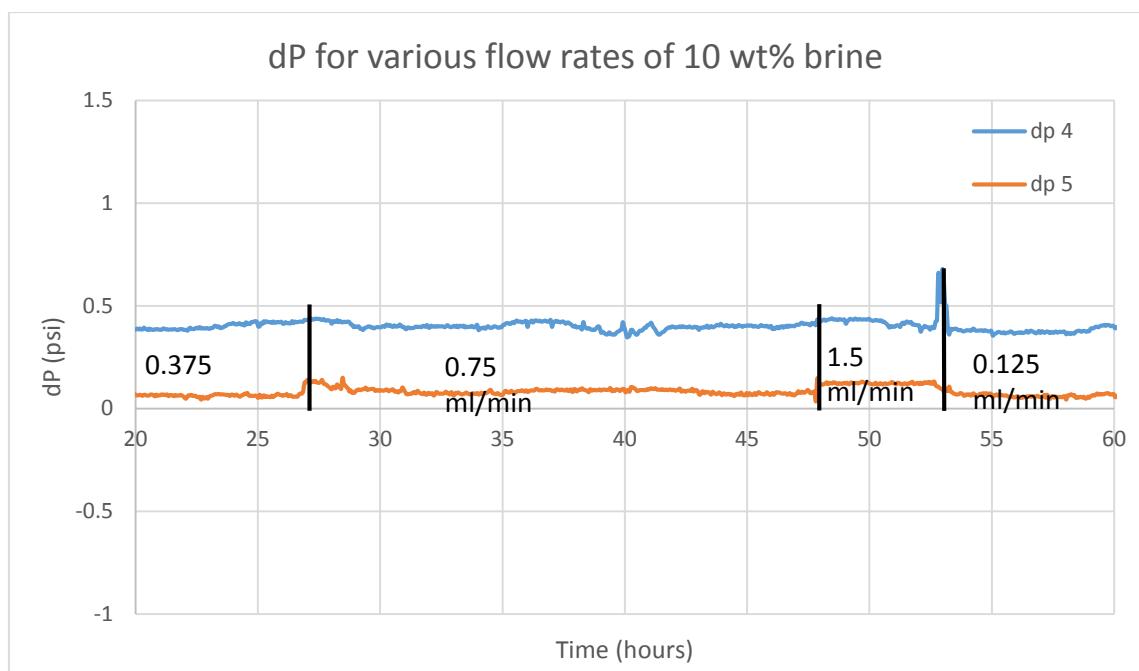


Figure 2.5. Pressure drop at different flow rates showing no hydrate blockage.

In order to fully solve this problem and prevent any blockage, we will further increase the salinity of the brine in the transducer lines. Since this is a dynamic system and hydrate is constantly forming/dissociating, we need to make sure we are operating precisely at three phase stability so no additional hydrate is formed or dissociated.

We have also noticed hydrate formation and blockage with gas injection. Although the system should be fully saturated with three phase brine, there is hydrate forming in the gas inlet to the core which prevents us from injecting gas. We have been able to solve this problem by heating the inlet to destroy the hydrate and allow gas to flow. Additionally, we have increased the pressure of the gas to break through any hydrate that temporarily forms in the inlet line when the gas first comes into contact with brine/water.

Next Steps

Once we have adequately solved the hydrate blockage and formation/dissociation problem, we will repack the sample and begin a new experiment with our updated procedure. We believe that with our small changes, we can collect data to construct a three phase relative permeability curve.

[Link to Phase 2, Task 6, continued work on permeability](#)

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

We performed one hydrate dissociation experiment outside of the CT scanner. This dissociation occurred over 21 days and allowed for the observation of 44 pressure rebounds during sample shut-in ranging from 1 hours to 9 days. This experiment contained variable range of pressure drops that allow us to look at the pressure response across a scale of perturbations magnitudes. We also observed our longest pressure rebound to date. We have now completed our depressurization experiments performed on sands in which hydrate was formed through gas injection into a brine-saturated sample.

We now consistently observe mass balance (methane recovered during depressurization = methane consumed during formation) which achieves Milestone 1.D.

Full documentation will be 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

We have continued to interpret the data acquired through our depressurization in the CT scanner. During pressure rebounds we observe heterogeneous changes in bulk density in our sand packs that suggest buildup of gas due to dissociation (density decrease) in some regions of the core and either movement of water or hydrate reformation in other parts of the core (density increase)

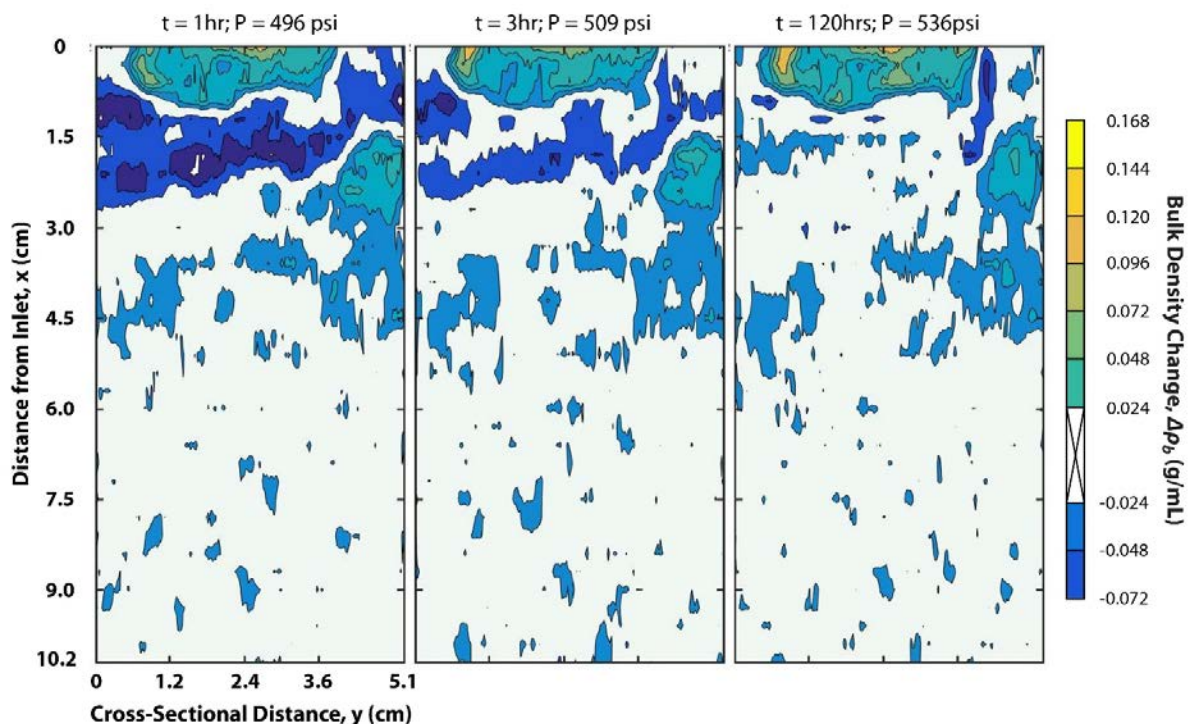


Figure 2. Changes in bulk density at 1 hour, 3 hours, and 120 hours after a pressure drop and release of gas. Blue shades represent a decrease in bulk density and green-yellow shades represent an increase in bulk density.

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

In this quarter, we conduct two experiments (Exp1 and Exp2) of methane hydrate growth in sandy sediments at excess gas conditions with different salinities and initial water saturations. In both experiments, we use X-ray micro-CT to monitor hydrate growth and clearly observe methane hydrate within sandy sediments.

Exp1 started from an initial methane gas pressure of 6.93 MPa, water saturation of 81% and a salinity of 0.6 wt% NaBr. We maintained the temperature at 5 ± 1 °C (see Figure. 4.1 a). The initial hydrate stability pressure at 5°C and an initial salinity of 0.6 wt% NaBr is 4.26 MPa. Methane hydrate immediately nucleated and methane gas pressure started to decrease (see Figure 4.1 b) as soon as the cooling started. Figure 4.1 shows the temperature and pressure evolutions during the first 3 days.

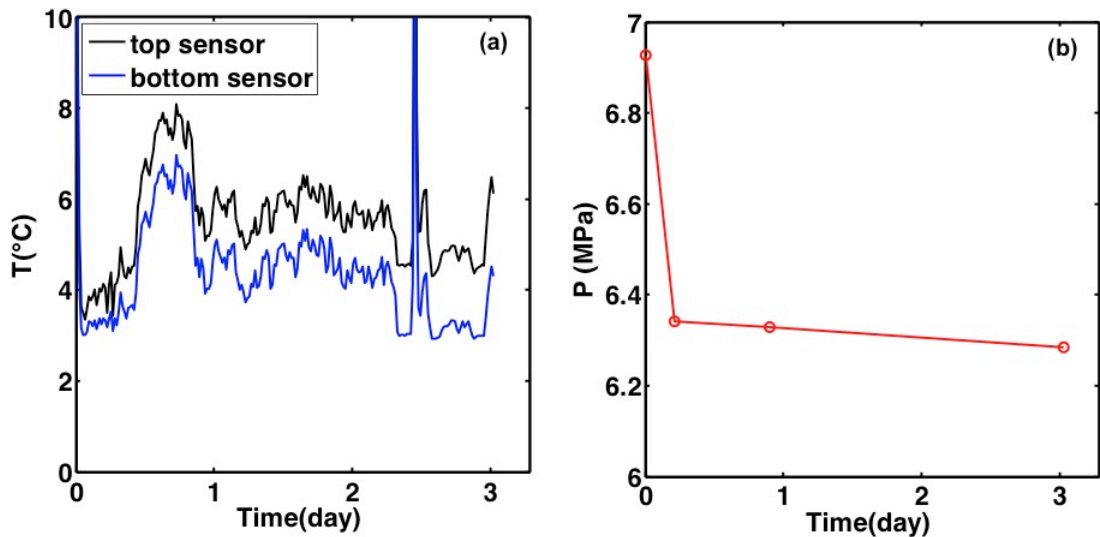


Figure 4.1 Temperature (a) and pressure (b) of Exp1 during the first 3 days. The initial methane hydrate stability pressure at 5°C and an initial salinity of 0.6 wt% NaBr is 4.26 MPa.

Figure 4.2 shows one original CT slice and its segmented analogue after 5 hours of methane hydrate growth. Similar to previous micro-CT observations on xenon hydrate bearing sand (Chen & Espinoza, 2018 - Fuel), methane hydrate also displays a porous structure, irregular shapes and heterogeneous distribution at the initial growth stage. The calculated hydrate saturation is 58.2 % and the porosity is 42.6 % in this particular region. In Exp1, there is difficulty in defining the brine phase and the hydrate phase.

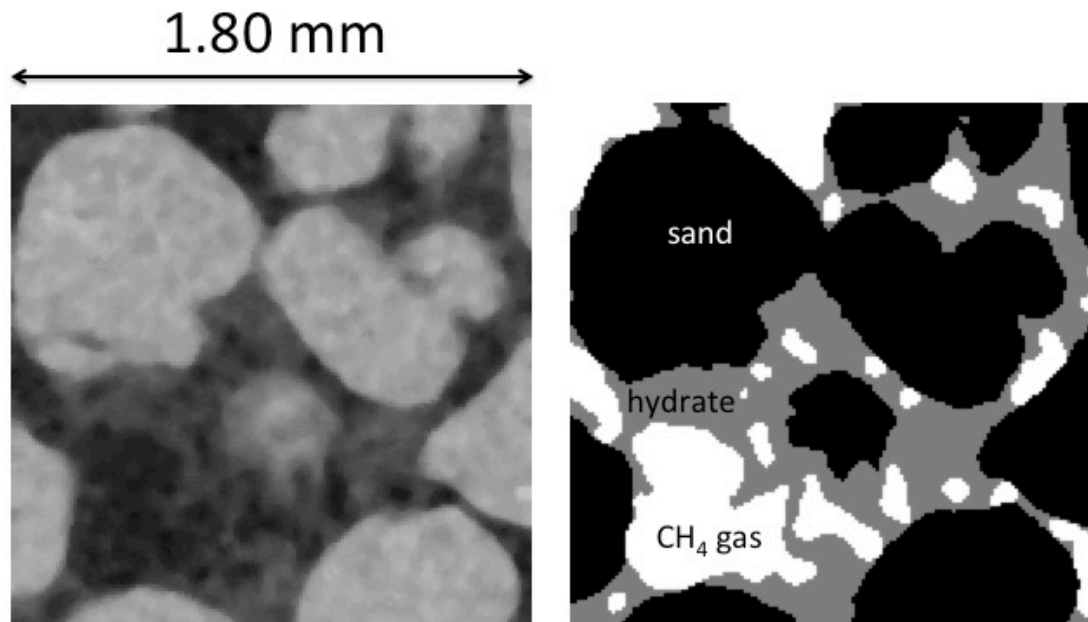


Figure 4.2 The original (left) and the segmented (right) CT slices of the sand at the same position after 5 hours of hydrate growth. In the segmented CT slice, black is sand grain, white is methane gas and the gray is methane hydrate. The porosity is 42.6% and the hydrate saturation is 58.2%. The image resolution is 12.0 μm .

Exp2 started from an initial methane gas pressure of 6.83 MPa, water saturation of 6.0 % and a salinity of 1.5 wt% NaBr. We maintained the temperature at 4.4 ± 1 °C. The initial hydrate stability pressure at 4.4°C and an initial salinity of 1.5 wt% NaBr is 4.09 MPa.

Figure 4.3 shows one slice of original CT and its segmented CT after 2 days of methane hydrate growth in sand. The upper-left quarter of the original CT (upper left) shows that the pore space formed by three sand grains is filled with a mixture of NaBr brine and methane hydrate. In CT imaging, methane hydrate has lower grayscale number than NaBr brine since NaBr is a stronger X-ray attenuating material than water and methane and methane hydrate does not contain NaBr within. Fig. 4.3 bottom shows the grayscale profile of the red arrow in the original CT (Fig. 4.3 upper left). The decrease in grayscale number indicates that, the outside of the water droplets has converted to methane hydrates, while the inside is still brine and more concentrated in NaBr. A threshold of 22000 (16-bit gray scale) is chosen herein to segment between brine and hydrate. The segmented CT image (upper right) shows the coexistence of four different phases, including, sand, brine, hydrate, and methane gas.

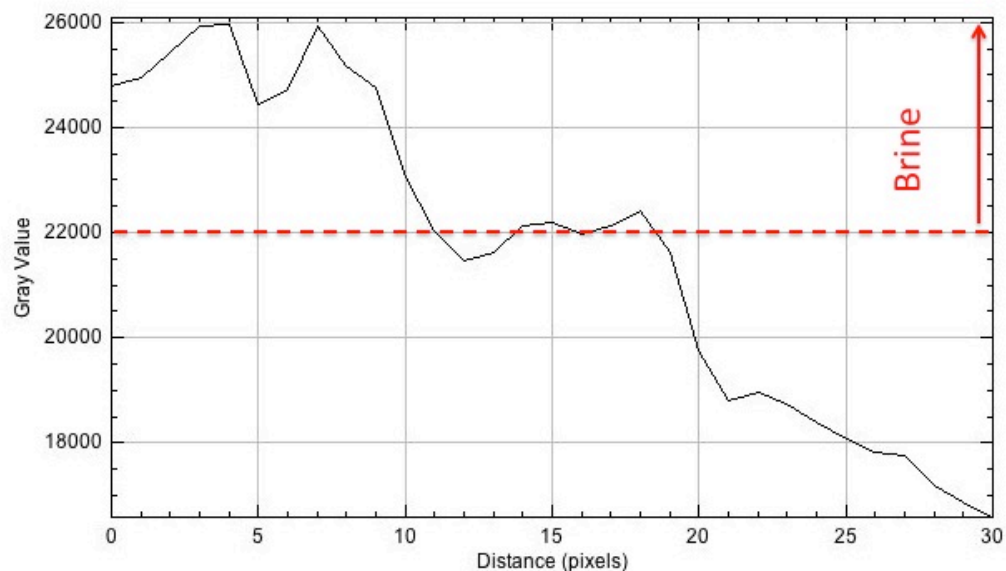
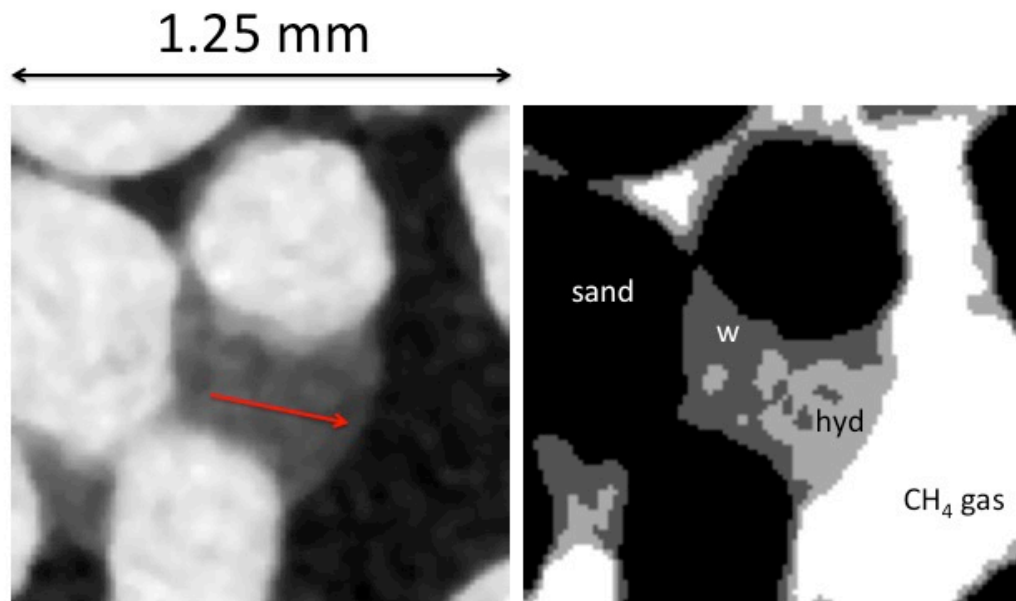


Figure 4.3 The co-existence of methane gas, methane hydrate and brine in sandy sediments after 2 days of hydrate growth in brine. Upper left: original CT of the sample; upper right: segmented CT showing sand as black, brine as dark gray, methane hydrate as light gray and gas as white; bottom, CT grayscale profile of the red arrow in original CT, which shows the inside of the pore is brine and the outside of the pore is methane hydrate with a threshold of 22000. Image resolution: 12.50 μm

Documentation of Milestone 1.F is to be included in 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 to be 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

We improved our data analysis technique and reprocessed the data from the previous experiments (Fig. 5.1-5.5). We started to characterize the GOM2 sediment samples using Raman spectroscopy. The sediment characterization will set the benchmark for our next experiments, in which we will synthesized methane hydrate in depressurized GOM2 sediments. Figure 5.2 shows the pressure and temperature data over time during the dissociation stage. Hydrate dissociation was initiated by decreasing pressure at 0.1 MPa steps. The temperature was maintained constant. The calculated corresponding equilibrium temperature at 3.07 MPa is 274.7 K, which is 3 K lower than the measured temperature. This offset may result from temperature measurement inaccuracy. To address this temperature inaccuracy concern, we made a new, improved insulation layer for the reaction chamber.

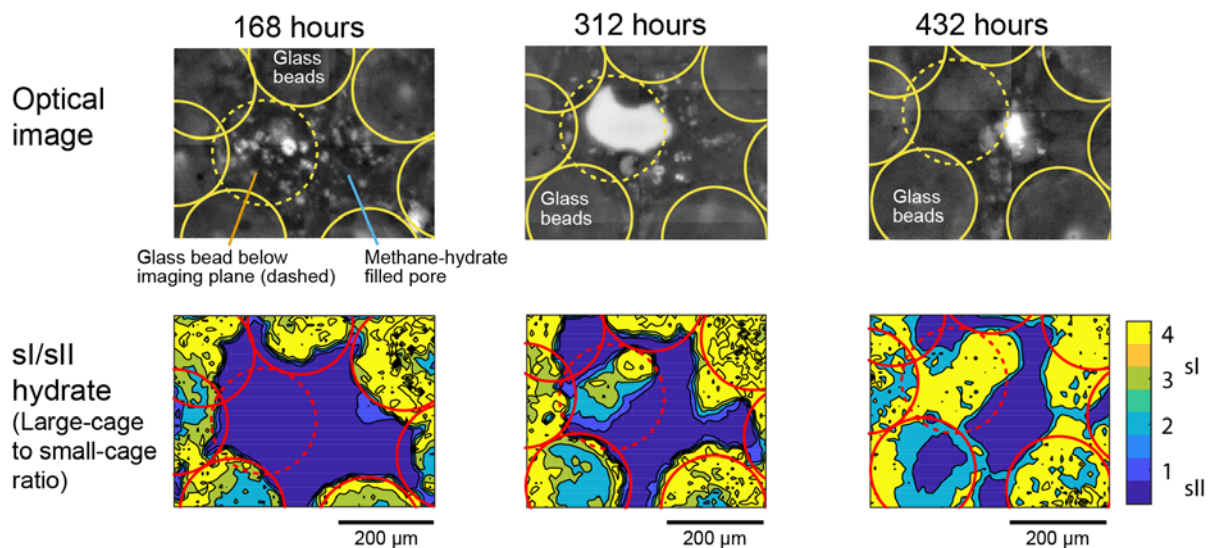


Figure 5.2 Hydrate formation: Optical images and Raman peak intensity ratios of large to small cages. Metastable sII to stable sI conversion initiated on grain surfaces and then migrated into the pore center. Stable sI hydrates attached to grain surfaces, and metastable sII hydrates in the pore space.

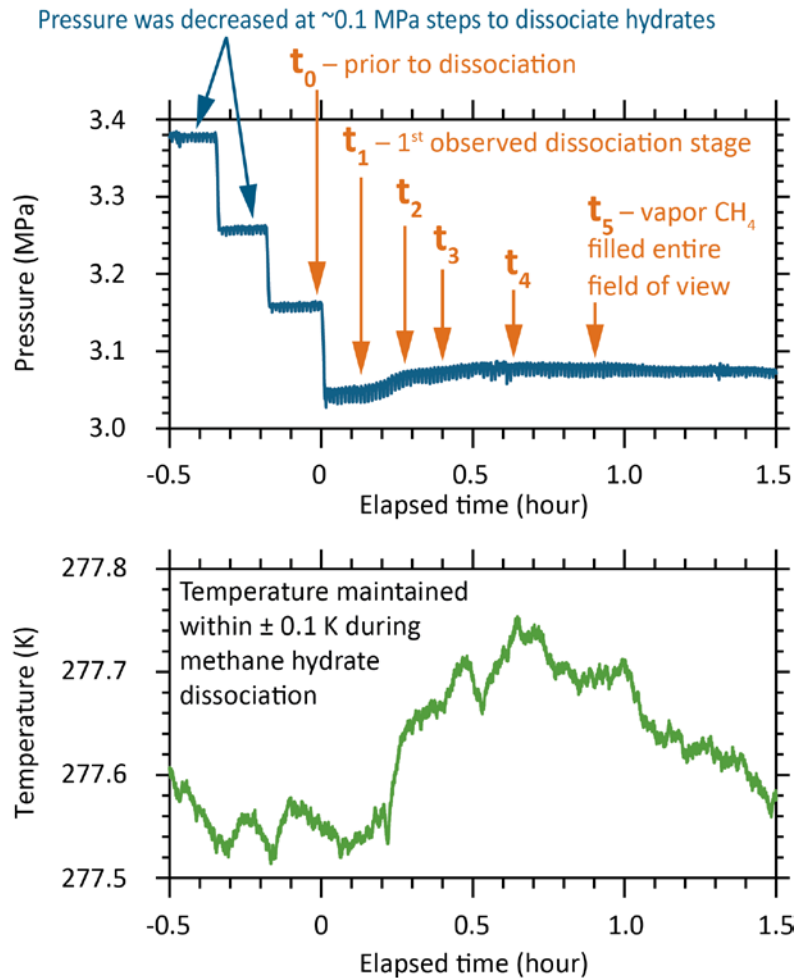


Figure 5.2. Pressure and temperature evolution during the dissociation stage. Time zero is aligned to the start of the hydrate dissociation. Hydrate dissociation was carried out by decreasing pressure at 0.1 MPa steps. The temperature was maintained constant. The dissociation of hydrate is characterized by the pressure in a constant volume chamber. Due to the small size of the sample, all hydrate dissociated after about an hour.

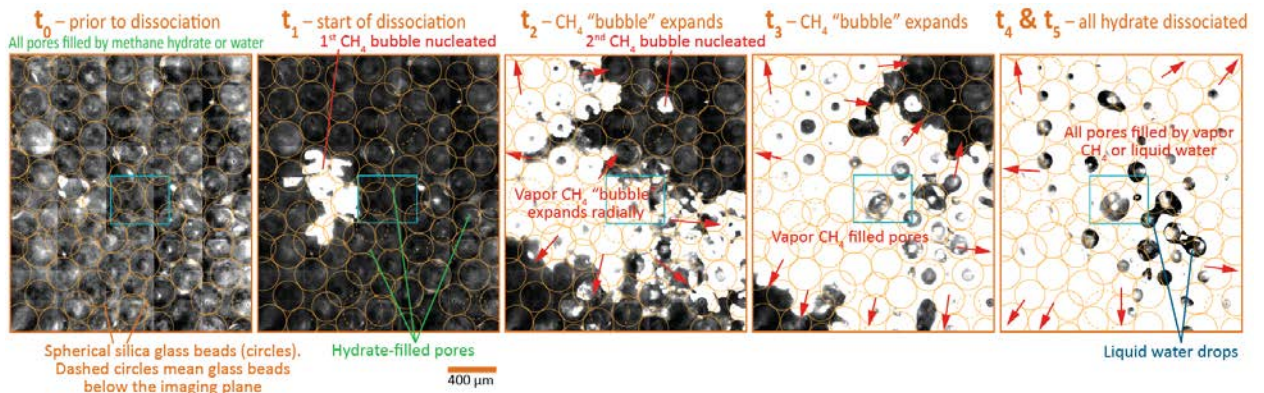


Figure 5.3. Hydrate dissociation: gas methane radial expansion in the pore network carrying out further dissociation. Dark region: methane hydrated filled pores. Bright region: vapor methane filled pores

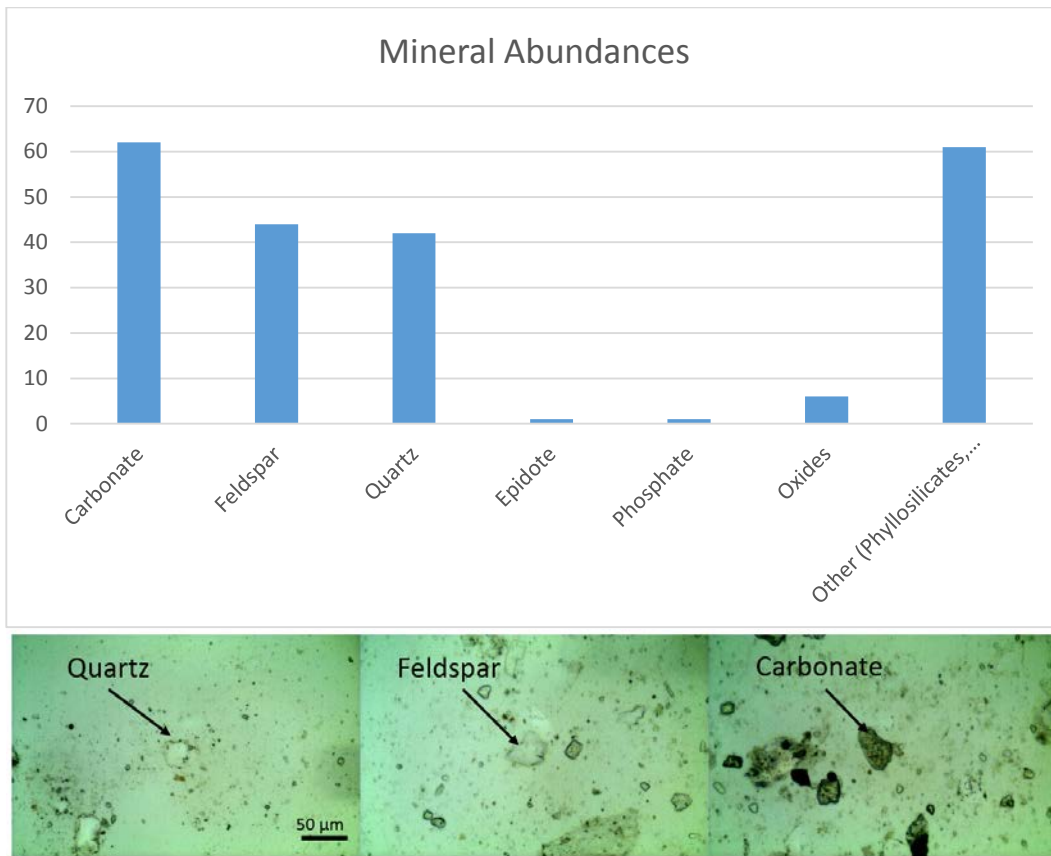


Figure 5.4. Mineral abundances characterized the Raman spectroscopy. The natural sediments are from core UT-GOM2-1-6FB-2.

Documentation of Milestone 1.H to be included in the 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

In the previous quarter, we have not obtained additional information to delineate the diffusion kinetics of methane release during dissociation. We are preparing an experiment to synthesize and dissociate methane hydrates in natural depressurized GOM2 sediments. Fig. 5.5 shows an updated figure of the pore-scale methane hydrate dissociation from the previous experiment. In a pore, methane hydrate dissociation initiated from the grain surfaces and then progressed into the pore center.

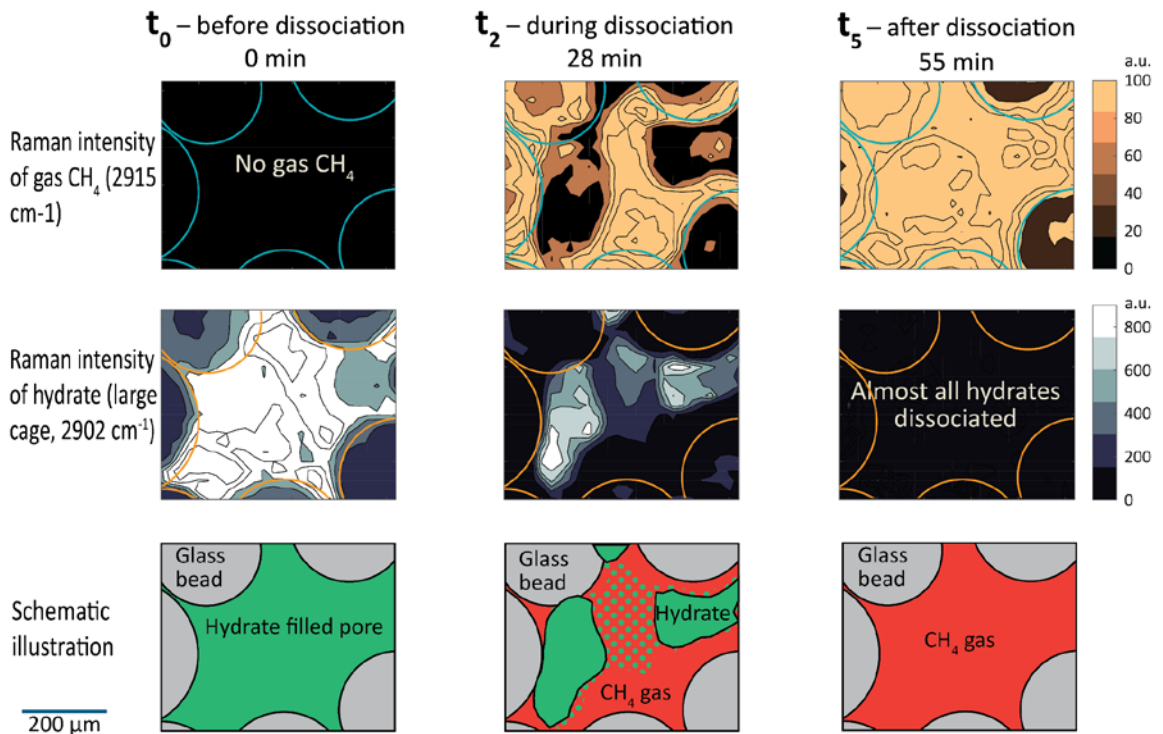


Figure 5.5. Hydrate dissociation initiated from the gain surfaces and then migrated into the pore centers.

[Link to Phase 2, Task 9, continued work on micro-Raman](#)

Decision Point: Budget Period 2 Continuation

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

FUTURE – 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.
[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: Not Started

[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: Not Started

[Link to actions for next Quarter, Task 7](#)

Subtask 7.2 Depressurization of intact pressure cores
Planned Finish: 9/30/19
Actual Finish: Not Started

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: Not Started

[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: Not Started

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: Not Started

[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: Not Started

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

Nothing to Report

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

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

Task 1.0 Project Management and Planning

Planned Finish: 09/30/19

Actual Finish: In progress

- Complete the Y2Q2 Quarterly
- Work on the Phase 1 Report

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: Complete

- Documentation of subtask completion in Y1Q4 Quarterly.
- Documentation of Milestone 1.B will be included in the Phase 1 report per the SOPO (Deliverable 1 for Task 2).

Subtask 2.2 Steady-State Permeability of Gas and Water of Sand-Pack Hydrate Samples

Planned Finish: 3/27/18

Actual Finish: Complete

- Full Documentation of Milestone 1.C will be included in the Phase 1 report per the SOPO (Deliverable 1 for Task 2).

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

Subtask 3.1 Depressurization Tests

Planned Finish: 6/27/17

Actual Finish: In progress

- Documentation of Milestone 1.D will be made in the Phase 1 report per the SOPO (Deliverable 1 Task 3).

Subtask 3.2 Depressurization Tests with CAT scan

Planned Finish: 3/27/18

Actual Finish: Complete

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: Complete

Documentation of subtask completion in Y1Q3 Quarterly. Documentation of Milestone 1.E is attached to the Y1Q3 quarterly and is to be included in the Phase 1 report per the SOPO (Deliverable 1 for Task 4)

Subtask 4.2 Micro-Scale CT Observations and Analysis

Planned Finish: 3/27/18

Actual Finish: Complete

- Documentation of Milestone 1.F will be included in the Phase 1 report per the SOPO (Deliverable 1 for Task 4)

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 to be included in the Phase 1 report per the SOPO (Deliverable 1 for Task 5)

Subtask 5.2 Micro-scale petrochemistry

Planned Finish: 03/21/18

Actual Finish: Complete

- Documentation of Milestone 1.H to be included in the Phase 1 report per the SOPO (Deliverable 2 for Task 5)

Subtask 5.2 Diffusion kinetics of methane release

Planned Finish: 03/27/18

Actual Finish: Complete

- Documentation of Milestone 1.H to be included in the Phase 1 report per the SOPO (Deliverable 2 for Task 5)

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: Not Started

- We will start this Task by 6/1/18.

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 1/1/19.

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: Not Started

- We will form hydrates using the formation method used in Task 2.0 to obtain hydrate saturations > 40% and then depressurize using the method we used in Task 3.0. This will allow us to view the pressure recovery behavior at higher saturations and in hydrate samples formed with different methods.

Subtask 7.2 Depressurization of intact pressure cores

Planned Finish: 9/30/19

Actual Finish: Not Started

- We will depressurize pressure core samples recovered during the UT-GOM2-1 Expedition. We will slowly depressurize these samples 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.

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: Not Started

- We will improve the CT image quality for the fine sediment from GOM2
- We will prepare the sample at different initial water saturation for hydrate growth

Subtask 8.2 Production Testing on GOM2 Samples Observed with Micro-CT

Planned Finish: 9/30/19

Actual Finish: Not Started

- We will design a similar but smaller vessel and cooling assembly to form methane hydrate in GOM2 sediment. This is because the GOM2 sediment mean grain size is roughly 1/20 of our previous used sand. Hence, the sample has to be much smaller and has to be very close to the X-ray gun to achieve good CT images.

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: Not Started

- We will synthesize and dissociate methane hydrates in natural depressurized sediments from the GOM2 project

Subtask 9.2 Micro-Raman Imaging of methane hydrate sandpacks

Planned Finish: 9/30/19

Actual Finish: Not Started

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>

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.

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
- <https://iq.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)

3. CHANGES/PROBLEMS:

This section highlights changes and problems encountered on the project.

a. Changes in approach and reasons for change

Nothing to Report.

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.

CURRENT - BUDGET PERIOD 1

By our calculations, we expect to be under 20% carry over from BP1 to BP2, once all charges for the final quarter of BP1 have posted.

FUTURE – 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				
Non-Federal Share	\$ 19,857	\$ 210,735	\$ 7,140	\$ 217,875				
Total Incurred Cost	\$ 127,073	\$ 605,928	\$ 161,898	\$ 767,826				
Variance								
Federal Share	\$ (2,032)	\$ (238,972)	\$ 65,022	\$ (173,950)				
Non-Federal Share	\$ 12,515	\$ 11,542	\$ (12,229)	\$ (687)				
Total Variance	\$ 10,483	\$ (227,430)	\$ 52,793	\$ (174,637)				

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								

National Energy Technology Laboratory

626 Cochrans Mill Road
P.O. Box 10940
Pittsburgh, PA 15236-0940

3610 Collins Ferry Road
P.O. Box 880
Morgantown, WV 26507-0880

13131 Dairy Ashford Road, Suite 225
Sugar Land, TX 77478

1450 Queen Avenue SW
Albany, OR 97321-2198

Arctic Energy Office
420 L Street, Suite 305
Anchorage, AK 99501

Visit the NETL website at:
www.netl.doe.gov



1-800-553-7681



U.S. DEPARTMENT OF
ENERGY

**NATIONAL ENERGY
TECHNOLOGY LABORATORY**