OIL & GAS

DOE Award No.: DE-FE-0028967

Quarterly Research Performance Progress Report (Period Ending 3/31/2017)

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:

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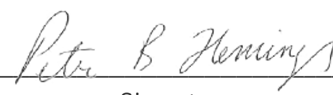

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National Energy Technology Laboratory

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Office of Fossil Energy

DISCLAIMER

1. ACCOMPLISHMENTS:

What was done? What was learned?

This report outlines the progress of the second quarter of the first year of the first budget period. The majority of the progress made was purchasing parts to build the laboratory equipment and beginning to test that equipment.

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	6/27/2017 (Y1Q3)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 2.1)	In progress
Milestone 1.C: Controlled and measured hydrate saturation using different methods	3/27/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 2.1)	
3 Milestone 1.D: Achieved depressurization and demonstrated mass balance	3/27/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 3.1)	
Milestone 1.E: Built and tested micro-consolidation device	6/27/2017 (Y1Q3)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 4.1)	In progress
Milestone 1.F: Achieved Hydrate formation and measurements in Micro-CT consolidation device	3/27/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 4.1)	
Milestone 1.G: Built and integrated high-pressure gas mixing chamber	3/27/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 5.1)	
Milestone 1.H: Micro-Raman analysis of synthetic complex methane hydrate	3/28/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 5.1)	
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?

<u>CURRENT- BUDGET PERIOD 1</u>

Task 1.0 Project Management and Planning

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

- The first Quarterly Report was submitted on February 6, 2017
- Work on the Website and Data Management Plan (DMP) are on-going.

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: In progress

- Continued purchasing equipment to build the laboratory apparatus.
- In order to get a sand sample into our core holder, we experimented with different methods of making a sandpack. The best results were obtained by preforming a moist sandpack in a plastic tube and freezing it. The saturation of the sand pack mixture could be directly controlled by the mass of water added. The frozen sand can then be extruded and maintain integrity while loading into the coreholder.

Subtask 2.2 Steady-State Permeability of Gas and Water of Sand-Pack Hydrate Samples
Planned Finish: 3/27/18
Actual Finish: Not Started

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

- We worked on a publication covering the freshwater dissociation experiment from the first quarter. The manuscript "Dissociation of laboratory-synthesized methane hydrate in coarse-grained sediments by slow depressurization":

Subtask 3.2 Depressurization Tests with CAT scan
Planned Finish: 03/27/18
Actual Finish: Not Started

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: In progress

During this quarter we made progress on building and testing the micro-consolidation device. Figure 1 shows the developed device together with the X-ray microtomograph used for imaging.

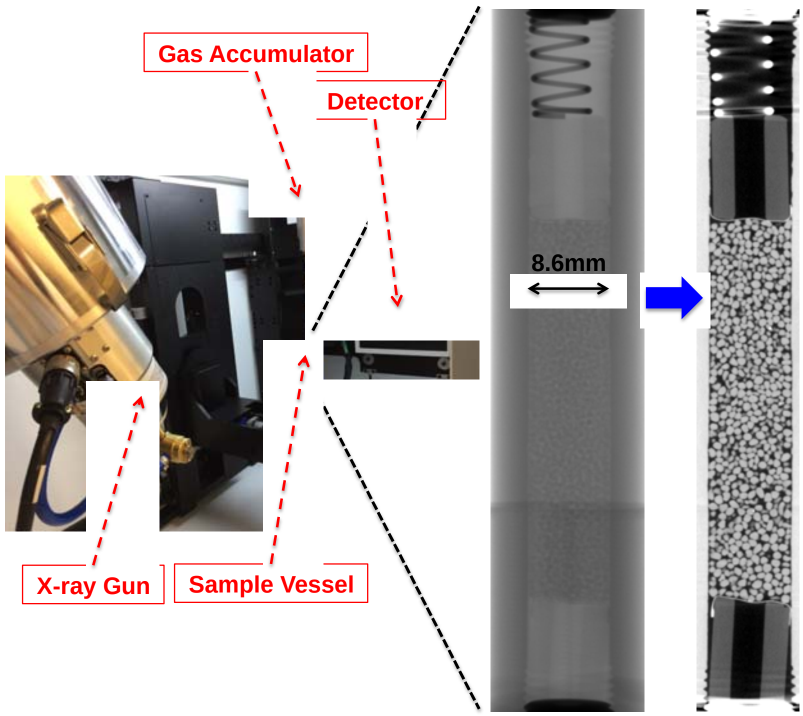

Figure 1. Micro-consolidation device mounted in a micro-CT scanner with a radiography (center) and a CT slice (right) of a sandpack sample. The apparatus consists of, from top to bottom, a large high-pressure vessel for storing gas, an analog gauge, a valve and the sediment micro-consolidation device itself. The sand pack bears an effective stress applied by a constant load spring.

We are currently working on additional developments for automatic/independent pressure/temperature control and measurement. These measurements will be implemented on an independent DAQ device that can be transferred from a temperature-controlled room to the CT machine. These upgrades do not limit the current operation of the device but offer improvements for long term monitoring. Upgrades are expected to be finished over the following quarter.

Subtask 4.2 Micro-Scale CT Observations and Analysis
Planned Finish: 03/27/18
Actual Finish: Not Started

During this quarter we have continued measurements with xenon hydrate and started bulk experiments with methane hydrate. We have improved the scanning resolution to better observe hydrate pore habit (Figure 2) and implemented volume segmentation techniques to calculate gas, water, and hydrate saturation. We utilize hydrate saturation from segmented images to calculate gas permeability with Corey-Brooks equations.

Figure 2 presents an example of xenon hydrate in a sandpack. Fig. 2a shows a CT slice after 4 days of gas injection within the stability zone. Fig. 2b shows calculated hydrate saturation profile along the sand pack. Experiments reveal high heterogeneity of hydrate saturation in a relatively homogeneous sandpack. Hydrate saturation in the top half of the sandpack is roughly 10% higher than the bottom half.

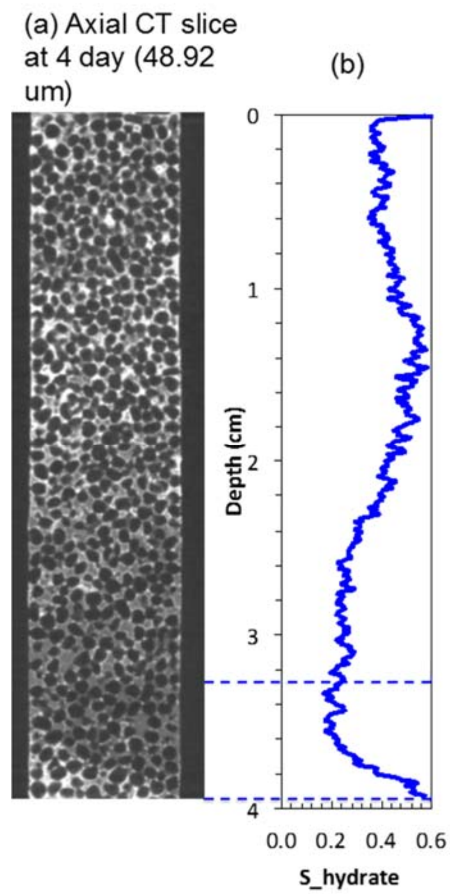

Figure 2. Heterogeneous hydrate distribution in sand and spatial variation of hydrate pore habits: a. an axial CT slice at 4 days; b. hydrate saturation profile in sand that shows heterogeneous hydrate saturation;.

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: In progress

During this quarter, we designed and built a "static" hydrate vessel for micro Raman analysis using Renishaw Micro-Raman system in Mineral Physics Laboratory at UT Austin. This vessel is designed to hold ~2 cm^3 of substrate, contains a sapphire window for in situ Raman analysis, and has a pressure capacity of 4000 psi. In addition, we have designed and built a thermoelectric chilling unit to bring the vessel down to as low as 0 degrees Celsius. We have outfitted this system with a 4000 psi pressure sensor, multiple thermistors, and a pressure control system (see Figure 3 below for the system). We have successfully synthesized methane hydrate at 7.7° C and 9.191 MPa (1333 psi) without porous media for the first time using the vessel, and took a series of Raman spectra of the methane hydrate crystals (Figures 4 and 5). Temperature and pressure were logged with a custom LabVIEW program integrated with the vessel and Raman system. In the following quarter, we will synthesize methane hydrate in porous media such as glass beads or natural sand particles under a relevant range of temperature and pressure conditions relevant to the methane hydrate reservoirs in the Gulf of Mexico.

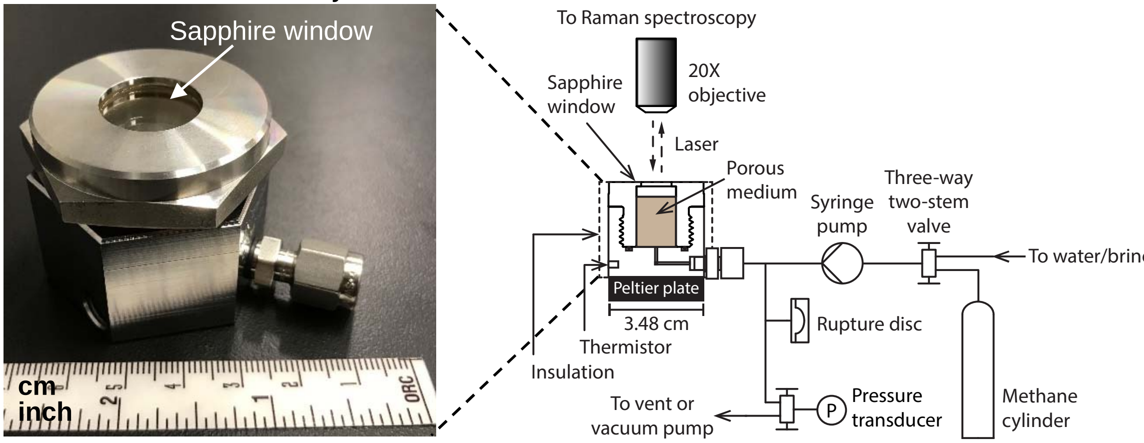

Figure 3. High-pressure and low-temperature vessel with an optical window for micro-Raman spectroscopic measurements of methane hydrates at UT Austin. The schematics on the right shows the integration of the vessel (on the left) with micro-Raman objective.

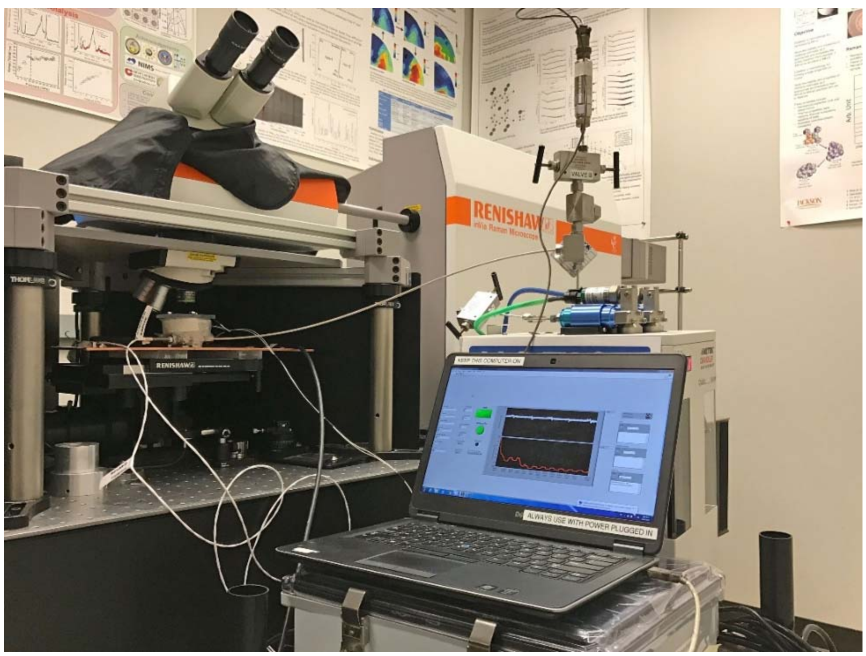

Figure 4. Synthetic methane hydrate in a pressure vessel coupled with Micro-Raman system at UT Austin. The Raman cell is placed under the Raman microscope for spectral examination. We have built a LabView Program in a portable computer to monitor the pressure and temperature of the sample chamber.

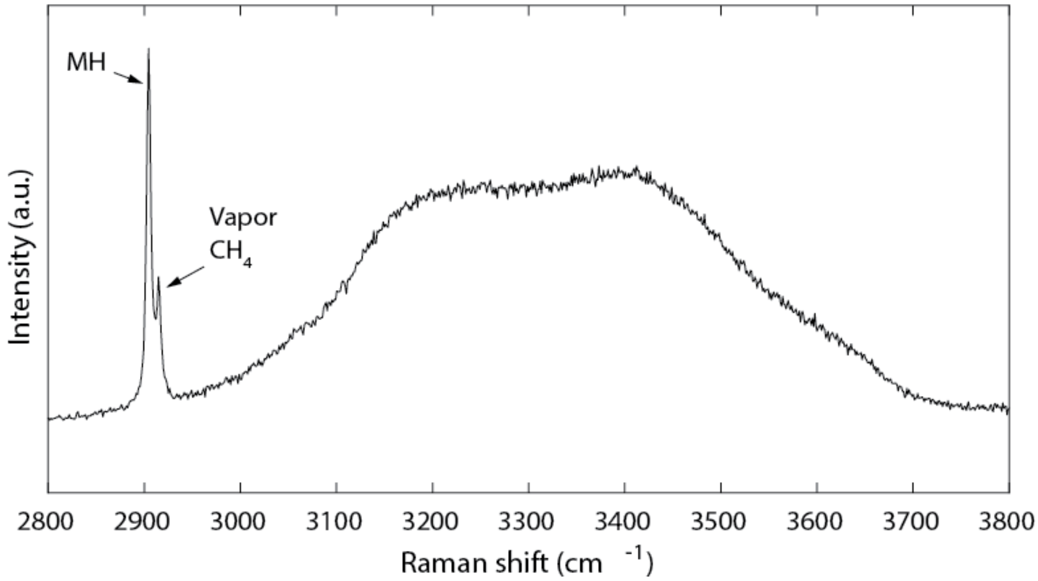

Figure 5. Raman spectra of methane hydrate (MH), vapor CH_4, and water measured by the Renishaw Raman system in a static pressure vessel. The broad peaks above 3000 cm-1 indicate water Raman bands. The methane hydrate was synthesized from vapor CH_4 and freshwater in the pressure vessel without porous media at 7.7° C and 9.191 MPa (1333 psi).

Subtask 5.2 Micro-scale petrochemistry
Planned Finish: 03/31/18
Actual Finish: Not Started

Subtask 5.2 Diffusion kinetics of methane release
Planned Finish: 3/27/18
Actual Finish: Not Started

Decision Point: Budget Period 2 Continuation

Nothing to report this period.

FUTURE – BUDGET PERIOD 2

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

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

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

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

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.
- An Abstract was prepared and submitted for a Poster to be presented at the 9th International Conference on Gas Hydrates, June 25-30, 2017, Denver, CO.

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

a. Task 1.0 Project Management and Planning

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

- Begin setting up an external project website

b. 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: In progress

- Assemble equipment and start performing hydrate formation experiments
- Obtain evidence of hydrate formation by comparing pressure drop for flow of brine through the sand pack

Subtask 2.2 Steady-State Permeability of Gas and Water of Sand-Pack Hydrate Samples
Planned Finish: 3/27/18
Actual Finish: Not Started

- We are currently optimizing our sample preparation methodology.

c. 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

- We will complete the manuscript "Dissociation of laboratory-synthesized methane hydrate in coarse-grained sediments by slow depressurization":
- Additional depressurization experiments in which we vary the magnitude of gas release at various stages of depressurization to test the pressure rebound due to salt diffusion to varying volumes of freshwater release will be delayed until the end of the DE-FE0023919 Hydrate Pressure Coring Marine Test.

Subtask 3.2 Depressurization Tests with CAT scan
Planned Finish: 3/27/18
Actual Finish: Not Started

- Nothing to Report.

d. 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: In progress

We will complete Milestone 1.E: “build and test micro-consolidation device

Subtask 4.2 Micro-Scale CT Observations and Analysis
Planned Finish: 3/27/18
Actual Finish: Not Started

Continue with methane hydrate growth and monitoring. Analysis will add computational fluid dynamic calculations to determine the influence of pore-habit and spatial variability on permeability of hydrate-bearing sand.

e. 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: In progress

- During the next quarter we will complete Raman analysis on hydrate formed in a glass bead substrate. After placing the glass bead substrate in the vessel, we will vacuum the system, flood the system with high-pressure methane, and then push brine into the system. Once cooled into the hydrate stability zone (<10 degrees Celsius), we will use Raman spectroscopy to map the hydrate saturation and pore fluid chemistry as a function of time and space. We will repeat this test several times using substrates of different diameters, before transitioning to natural sand sediment.

Subtask 5.2 Micro-scale petrochemistry
Planned Finish: 03/21/18
Actual Finish: Not Started

- Nothing to Report.

Subtask 5.2 Diffusion kinetics of methane release
Planned Finish: 03/27/18
Actual Finish: Not Started

- Nothing to Report.

2. PRODUCTS:

What has the project produced?

Research Performance Progress Report (Period ending 12/31/16)

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 to be presented at the 9th International Conference on Gas Hydrates, June 25-30, 2017, Denver, CO.

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

Nothing to Report.

c. Technologies or techniques

Nothing to Report.

d. Inventions, patent applications, and/or licenses

Nothing to Report.

e. Other products

Nothing to Report.

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

There have been significant delays in purchasing, manufacturing, and integrating our experimental systems. In addition, on graduate student has left unexpectedly. We have recruited an additional graduate student and are looking forward to a productive summer.

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

Task 1 – Project Management Plan

- Data Management Plan in progress.

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				
Non-Federal Share	$ 10,800	$ 10,800	$ 10,800	$ 21,600				
Total Incurred Cost	$ 17,549	$ 17,549	$ 61,703	$ 79,252				
Variance								
Federal Share	$ (276,748)	$ (276,748)	$ (31,135)	$ (307,883)				
Non-Federal Share	$ (159,663)	$ (159,663)	$ 3,671	$ (155,993)				
Total Variance	$ (436,411)	$ (436,411)	$ (27,465)	$ (463,876)				

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		07/01/16-09/30/18	
	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								
Non-Federal Share								
Total Incurred Cost								
Variance								
Federal Share								
Non-Federal Share								
Total Variance								

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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