NATIONAL ENERGY TECHNOLOGY LABORATORY

OIL & GAS

DOE Award No.: DE-FE-0028967

Quarterly Research Performance Progress Report (Period Ending 12/31/2016)

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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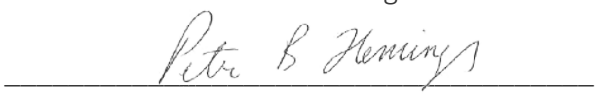

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Office of Fossil Energy

1. ACCOMPLISHMENTS:

What was done? What was learned?

This report outlines the progress of the first quarter of the first year of the first budget period. The majority of the progress made was setting up project management and the project Kick-Off Meeting.

A. What are the major goals of the project?

The goals of this project are to provide a systematic understanding of relative permeability and dissipation behavior in primarily 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	5/29/2017 (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	2/27/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 2.1)	
3 Milestone 1.D: Achieved depressurization and demonstrated mass balance	2/27/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 3.1)	
Milestone 1.E: Built and tested micro-consolidation device	5/29/2017 (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	2/27/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 4.1)	
Milestone 1.G: Built and integrated high-pressure gas mixing chamber	2/27/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 5.1)	
Milestone 1.H: Micro-Raman analysis of synthetic complex methane hydrate	2/28/2018 (Y2Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 5.1)	
Milestone 2.A - Measurement of relative permeability in sand-pack cores.	12/7/2018 (Y3Q2)		Documentation of milestone achievement within required project reporting / deliverables (Deliverable 6.1)	
Milestone 2.B - Measurement of relative permeability in intact pressure cores.	9/27/2019 (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.	9/27/2019 (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.	9/27/2019 (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;	12/7/2018 (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;	9/27/2019 (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: 09/30/18
Actual Finish: In progress

- The Project Investigator dedicated Project Manager for this project
- A Project Management Plan was created
- The Kick-Off Meeting was held on November 22, 2016
- Development of SharePoint site

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

- PhD student Dong See Hong has started working with Daigle and DiCarlo
- Commenced work on building sand pack, forming hydrate

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 ran a depressurization experiment in which methane hydrate was formed in a sand pack saturated with freshwater. This experiment provided a baseline of pressure rebound in response to perturbations in the absence of salt diffusion.

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 have designed an aluminum pressure vessel for X-ray monitoring and tomography (Figure 1). The sediment is packed according to "k_o" consolidation conditions with no lateral displacement and constant vertical axial stress applied with a spring. Figure 1 a shows the setup picture: (1) the k_o vessel has an inside diameter of 0.88 cm and an inside length of 7.26 cm, and is packed with sand at irreducible water saturation, (2) the top accumulator vessel is filled with gas, and (3) high-pressure valves connect the two vessels. We have pressure tested the vessel and already done preliminary hydrate experiments on it with success. The device is currently being updated to have an independent DAQ system for pressure and temperature control and measurement.

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

Before using methane to form hydrate in sandpack, we choose Xenon as an analogue since Xenon can form hydrate at room temperature. At a room temperature of 23°C, the experiment begins with opening the valves to allow Xenon force into the bottom vessel at 2.52 MPa, which is 0.95 MPa higher than the hydrate equilibrium pressure of 1.57 MPa. Figure 1 b shows the micro X-ray radiography image of the bottom vessel at 38 days. The image is taken at a resolution of 49.79 um. In this image, dark color means more X-ray attenuation. Xenon hydrate has a large X-ray attenuation comparable almost to that of stainless steel (NIST). We can see hydrate forms inside the top spacer and the sandpack and nearby the spring. Figure Xc shows the reconstructed orthogonal micro-CT image of the bottom vessel, in which white color means more X-ray attenuation. Similarly, we see hydrate concentrating in the top spacer and nearby the bottom spring, and we can see variations of hydrate saturation along the sandpack axial directon. Ongoing work studies the hydrate growth kinetics in detail and then start with methane hydrate experiment in sandpack.

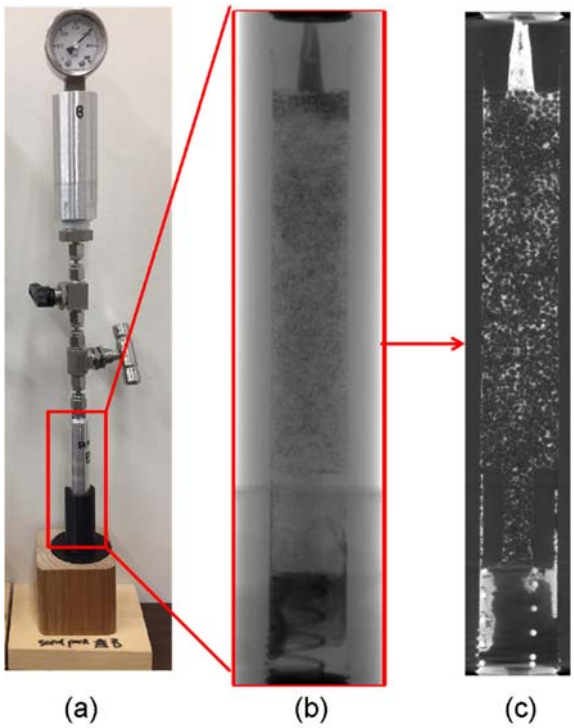
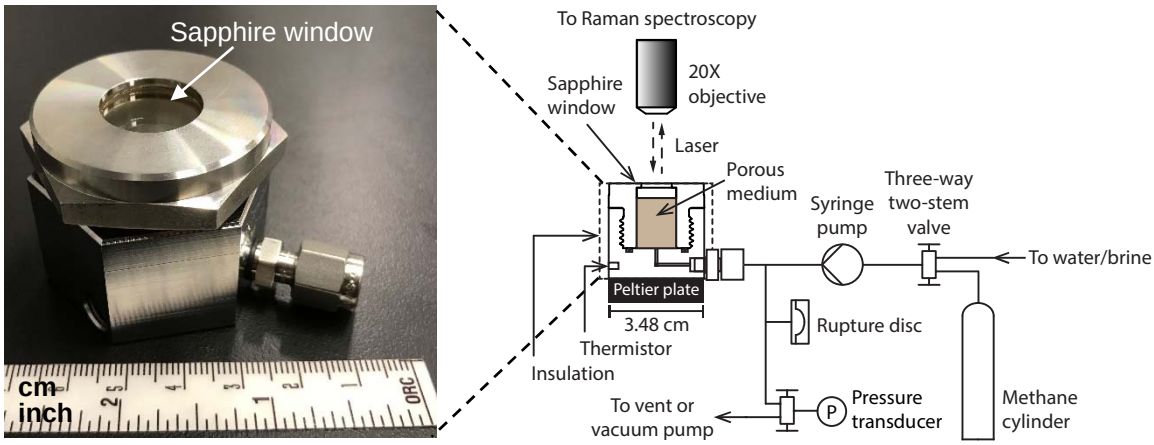


Figure 1. (a) Hydrate experiment set-up picture, (b) X-ray radiography image of the k_o pressure vessel at 38 days (resolution = 49.79 um, dark color is hydrate or stainless steel), and (c) micro CT image slice of the k_o pressure vessel at 38 days in orthogonal view (resolution = 49.79 um, white color is hydrate or stainless steel)

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 controls system. We will log temperature and pressure with a custom LabVIEW program we designed and tested this past quarter.



Subtask 5.2 Micro-scale petrochemistry
Planned Finish: 03/31/15
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.

<u>FUTURE – BUDGET PERIOD 2</u>

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?

Nothing to Report.

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

- Complete the Data Management Plan
- 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

- Nothing to Report

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 plan to run 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.

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

- Will make the switch from Xenon hydrate as an analogue to methane hydrate experiments

Subtask 4.2 Micro-Scale CT Observations and Analysis

Planned Finish: 3/27/18
Actual Finish: Not Started

- Nothing to Report.

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?

- The project produced a Kick-Off Presentation communicating the project management and technical plans forward.
  - This was shared with the Department of Energy during the Kick-Off Meeting on November 22, 2016.
- The project produced a Project Management Plan

a. Publications, conference papers, and presentations

Nothing to Report.

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

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

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						
Non-Federal Share	$ 10,800	$ 10,800						
Total Incurred Cost	$ 17,549	$ 17,549						
Variance								
Federal Share	$ (276,748)	$ (276,748)						
Non-Federal Share	$ (159,663)	$ (159,663)						
Total Variance	$ (436,411)	$ (436,411)						

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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