NATIONAL ENERGY TECHNOLOGY LABORATORY
OIL & GAS

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Quarterly Research Performance Progress Report)

(Period Ending 09/30/2017)

PROPERTIES OF SEDIMENTS CONTAINING METHANE HYDRATE, WATER, AND GAS SUBJECTED TO CHANGING GAS COMPOSITIONS

Project Period (April 1, 2012 to open)

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U.S. DEPARTMENT OF ENERGY | NATIONAL ENERGY TECHNOLOGY LABORATORY

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RESEARCH PERFORMANCE PROGRESS REPORT

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ACCOMPLISHMENTS:

Subtask 10 b Vessel modification – The new LBNL hydrostatic hydrate vessel was manufactured to meet the new design criteria, allowing improved investigation of gradients on hydrate behavior with improved safety. A commercial vendor manufactured the vessel and delivered it after numerous delays in manufacture including one that required remanufacturing the vessel due to non-matching threads.

Status: COMPLETE

In FY17Q3 we received the vessel, modified the pressure vessel end caps to allow standard tubing to pass through. We ordered fittings and tubing to finish building out the system and researched and ordered best available sample sleeves for hydrate. We designed/specified new custom endcaps for samples and got them in the queue for manufacture.

Task 11. Assessment of thermal gradient modification methods and Investigation of the effect of thermal gradient and gradient oscillation on hydrate behavior – We are designing methods to temperature gradient application. We are investigating the use of viscosity adjustment chemicals (gelatin) to reduce fluid convection and found the correct formula resulting in the proper viscosity, the use of

different heating/cooling coils as the new vessel has larger ports to allow this, and the use of different heating/cooling techniques including electric heaters on the outside of the vessel which have been sized and purchased. We experimentally determined that the selected heaters are sufficiently X-ray transparent and do not affect the images. We have yet to form methane hydrate and alter the thermal gradient or apply oscillating gradients and these studies will be initiated upon completion of fitting out the new vessel in Q4.

Status: In Q4, we investigated a number of methods to induce a thermal gradient. These include the use of a cartridge heater (rejected because of geometry and custom manufacture cost), electric sheet heater (was not as effective as envisioned), a warm flow loop (still under consideration, but initial tests showed less-than-expected performance, a custom low voltage nichrome wire heater (still under consideration, however there are safety considerations). An additional technique to be considered is the use of low-voltage small ink-jet cartridge heaters placed in a thermowell-like tube at the end of the vessel. As can be seen from the photo below, 2 x ¼ inch pass throughs are available which could contain the ink-jet heaters, an improved warm-water tube, or a nichrome wire assembly.

Tests this quarter were performed in the older vessel because of sealing problems with the cooling jacket on the new vessel. These problems required redesigning the cooling jacket and modifying the application of the jacket. A weak gelatin was used as the confining fluid to improve conductive heat transfer, and temperature measurements were made using a sliding thermocouple in a Teflon fitting.

Focus next quarter: Improve thermal gradients in new vessel while observing hydrate formation/dissociation.

Task 12. Investigation of the hydrate dissociation point in saline systems with respect to gas production rate. –We investigated the water solid/liquid transition in brine as an analog for methane hydrate in brine by monitoring the temperature of the melting solid phase at its surface and interior as it melts, both slowly, and rapidly, both in the pure phases and in porous media. Several different iterations of the thawing experiment were done to model the phase change conditions at the interface between ice (analog for hydrate) and 8% NaCl, with the question being did the ice melt (dissociate) at 0C (normal equilibrium point for zero salt) or at the temperature expected for the salt water mixture. The ice, in both crushed and block initial condition, was placed in 5C bath of 8 %NaCl, in either mixed (representing slow dissociation – transport fast compared to phase change) or unmixed (modeling fast dissociation where transport is slow compared to dissociation). Results showed that the ice stayed at 0C but as soon as the surface was exposed to the salt water, the temperature was the same as the salt water, which rose in temperature at a steady rate until reaching room temperature. If the bath was mixed the main difference was that the warming in the mixed system was faster. Because of potential errors in the test, a second setup was to designed to minimize the mixing of the meltwater and saltwater. A block of ice was placed on sand saturated with -5C 8% NaCl. Thermocouples were frozen into the ice, held on the interface between the sand and the ice, and within the sand. The ice thermocouple stayed at 0C until melting. The thermocouples at the interface were very sensitive to exact positioning, but if they maintained direct contact with the ice they behaved like the ice block behaved. If they were in contact with the sand, they followed the more gradual temperature increase of the 8%NaCl. In summary, the salt water did not appear depress the melting point of pure ice at the interface for slow melting (fast dissociation), whereas the salt affected the melting when mixed (slow dissociation). By analogy, gas hydrate should be affected by the salt present when transport is fast, and when transport is slow, it should be less impacted.

Status: COMPLETE

Task 13. Continued Studies of Hydrate-Bearing Layered Systems

This task was put on hold until the new vessel was fitted out and will be initiated in FY18Q1. Some of the materials needed have been obtained for use in the tests.

Status: Initiated but incomplete. Discussions with modelers will improve experiment direction.

Focus next quarter: Build initial layered hydrate tests in new vessel. New vessel is nearly operational.

Task 14. Continued Computational Rock Mechanics

Regarding prediction of the behavior of a hydrate-bearing medium based on X-ray micro CT and three hydrate habits we have a working version of a new permeability code going and can run it on our hydrate volumes. We are still moving towards full 389 pixel cubes for all three habits (grain cementing, pore filling, and grain coating). Progress has been slow due to availability of the appropriate researcher.

Status: Initiated but incomplete. No change from Q3.

Focus next quarter: Diversify pool of researchers available to perform task and run simulations on permeability while reexamining geomechanical behavior.

Task 15. Experimental work in response to current challenges

No progress this quarter.

Focus next quarter: Continue focus on layered systems. Future focus on varying brine composition in layered and non-layered systems.

Task 16. Continued investigation of layered systems

Evaluation of sands has initiated for this task. The pressure vessel is functioning and major equipment is available.

Focus next quarter: Setup for this task will begin in FY18Q1.

Task 17. Comparison of the effect of vertical and horizontal wells for gas production in a layered hydrate-bearing system

Discussion of modeling/experiment strategies has begun. Devices needed within the sample to initiate the dissociation are being designed.

Focus next quarter: Experiments are expected to begin in FY18Q2.

Milestone Table

Milestone Title	Milestone Description	Planned Completion Date	Actual Completion Date	Status / Results
Report on Layered Hydrate	A report describing the data collected in our first layered hydrate test will be submitted.	January 31, 2015 Delayed – expected by September 30, 2015	Included with quarterly update 5/05/16	Complete
Go/NoGo on vessel	A brief letter report will be submitted following Task 10a to inform the DOE of the go/no go decision on vessel remanufacture. (Tasks 10).	September 30, 2015	Informally contained in update 11/16	Complete
Topical Report	A report documenting the results of laboratory tests examining the effects of thermal gradients and gradient oscillation on hydrate behavior will be submitted. (Task11)	March 31, 2016	Delayed	Will resume in new vessel in Q4
Topical Report	A report documenting the results of laboratory tests investigating the gas hydrate equilibrium point versus the gas production rate will be submitted. (Task 12)	June 30, 2016	June 30, 2016	Laboratory work complete and powerpoint presentation assembled. Letter report in preparation.
Topical Report	A report documenting the results of laboratory tests on layered systems.	July 31, 2017	Delayed	Work will resume in Q4
Grain-scale Computation of Hydrate-Bearing Sand Properties Based on micro CT Sample Description	A report documenting the results of numerical simulations on multigrain scale flow and mechanical simulations.	May 31, 2017	Delayed, data fields generated and shared with NETL	Simulations will resume in Q4
Topical Report	A report documenting the results of current challenge laboratory tests and their interpretation.	July 31, 2017	Delayed	Experimental work will resume in Q4 with new vessel
Topical Report	A report adding to the observations on layered systems	January 31, 2018		
Topical Report	An experiment report documenting the results of gas production from laboratory-simulated vertical and horizontal wells.	July 31, 2018		
Conference Papers/	Documents include conference papers,	Minimum of 7 business days		

Proceedings/ Articles	proceedings, presentations, journal articles, and press releases.	prior to submission		

PRODUCTS:

None to report this quarter.

CHANGES/PROBLEMS:

As mentioned in the accomplishments section, several tasks were delayed pending the arrival of the new pressure vessel. Several additional delays were caused by the lack of availability of key personnel. The new vessel has been approved, manufactured by a reputable pressure manufacturer, pressure tested, and delivered. Testing showed severe problems sealing the temperature jacket. Several designs were evaluated, and minor modifications were made and a new jacket was built and testing is complete.

Personnel problems have been alleviated by redirection of personnel tasks and the arrival of a graduate student in September, and the increased availability of critical researchers. The new graduate student is currently in training. We have also obtained some availability from an experimental post-doctoral researcher with experience in controlled pressure/temperature core flood work wishing to broaden his experience.

SPECIAL REPORTING REQUIREMENTS:

NA

BUDGETARY INFORMATION:

Actual Cost (this quarter)	Actual Cost (cumulative for BP)	Funds available (for the BP)	Balance of unspent funds (for the BP)	Actual Cost (cumulative for the full FWP)	Funds available (for the full FWP)	Balance of unspent funds (for the full FWP)
$28,905	$32,876	$305,000	$172,124	$487,171	$685,000	$197,829

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