

DOE Award No.: ESD12011

Quarterly Research Performance Progress Report)

(Period Ending 06/30/2017)

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

Project Period (April 1, 2012 to open)

Submitted by:

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**NATIONAL ENERGY
TECHNOLOGY LABORATORY**

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RESEARCH PERFORMANCE PROGRESS REPORT

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: Initiated but incomplete.

Focus next quarter: Induce thermal gradients in new vessel.

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 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 Q4. Some of the materials needed have been obtained for use in the tests.

Status: Initiated but incomplete.

Focus next quarter: Build initial layered hydrate tests in new vessel.

Task 14. Continued Computational Rock Mechanics

Regarding prediction of the behavior of a hydrate-bearing medium based on X-ray microCT 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.

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.

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 microCT 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

PRODUCTS:

Kneafsey, T.J. and S.E. Borglin, Experimental Observations of Methane Hydrate Dissociation in Layered Media, full paper, Ninth International Conference on Gas Hydrates, Denver Colorado, June 2017

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 inavailability of key personel. The new vessel has been approved, manufactured by a reputable pressure manufacturer, pressure

tested, and delivered. It will allow for larger samples, easier test assembly, and more measurement ports. It is currently being outfitted for temperature control, end pieces, and sleeves, and will be in use very shortly. Personnel problems will be alleviated by the arrival of a graduate student in September, and the increased availability of critical researchers.

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)
\$46,245	\$103,971	\$180,000	\$76,029	\$458,266	\$560,000	\$101,734

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