

# Oil & Natural Gas Technology

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Quarterly Research Performance Progress Report (Period ending 06/30/2014)

## Hydrate-Bearing Clayey Sediments: Morphology, Physical Properties, Production and Engineering/Geological Implications

Project Period (10/1/2012 to 9/30/2016)

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## ACCOMPLISHMENTS

**Context – Goals.** *Fine grained sediments host more than 90% of the global gas hydrate accumulations. Yet, hydrate formation in clayey sediments is least understood and characterized. This research focuses on hydrate bearing clayey sediments. The goals of this research are (1) to gain a fundamental understanding of hydrate formation and ensuing morphology, (2) to develop laboratory techniques to emulate “natural” formations, (3) to assess and develop analytical tools to predict physical properties, (4) to evaluate engineering and geological implications, and (5) to advance gas production alternatives to recover methane from these sediments.*

### Accomplished

The main accomplishments for this period include:

- Completion of hydrate formation station
  - Design and fabrication of 2 new X-ray transparent aluminum chambers
  - Fabrication of frame for mounting the 3 aluminum hydrate formation chambers
  - Fabrication of chamber-to-CT scanner adapter plate
- Geometric calibration of X-ray CT scanner
- Formation of CO<sub>2</sub> hydrate in fine-grained sediment
  - Multiple conditions
- Evaluation of elastic strength and stiffness with varying hydrate lens angle

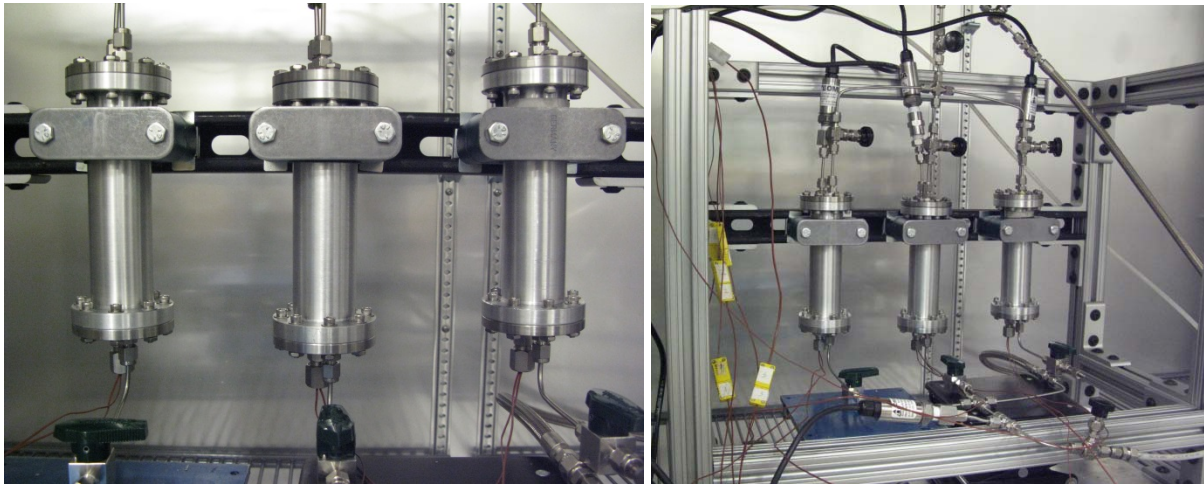
### Plan - Next reporting period

Experiment with different methods of forming gas hydrate in fine-grained sediments. Physical properties of hydrate-bearing fine-grained sediments: advance numerical solutions of large-strain stiffness and strength of various hydrate lens morphologies with elastoplastic materials.

## Research in Progress

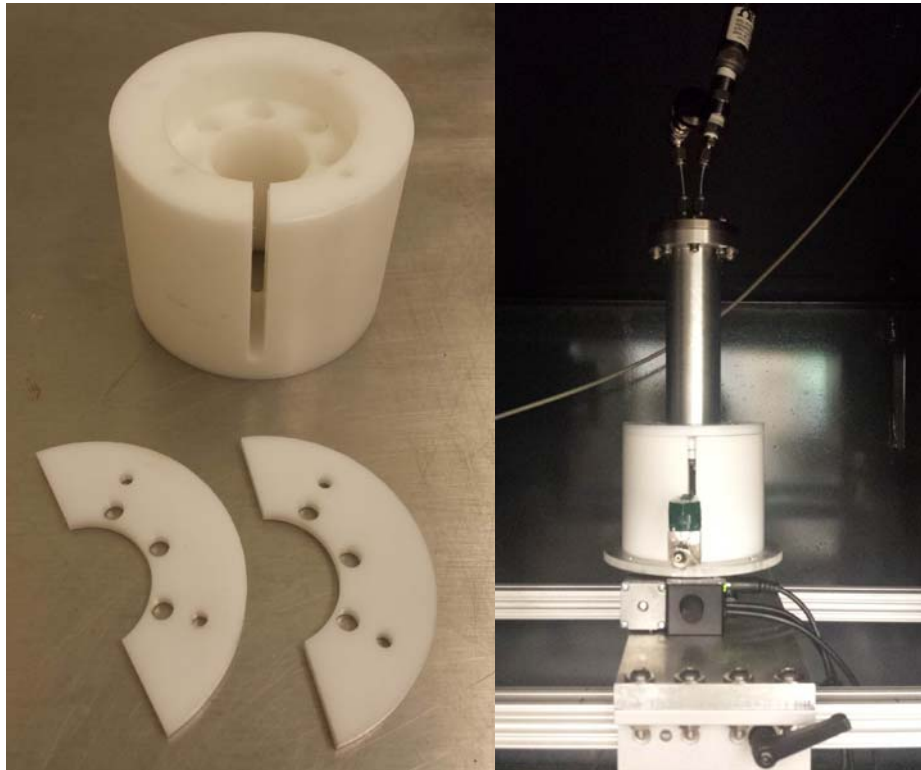
### Complete hydrate formation equipment

Two additional X-ray transparent, aluminum chambers were fabricated to allow for three, simultaneous hydrate formation experiments. The design of the new chambers is identical to the first fabricated chamber design, except for a new port modification for the steel end caps. A stand was constructed from t-slotted extruded aluminum to mount all chambers and peripherals.



Additionally, an adapter was made from white Delrin plastic to attach the aluminum chambers to the rotary stage in the X-ray CT scanner. This adapter allows the chambers to be scanned with minimal movement and to be placed in the same position through multiple scans, which allows for image comparison studies.

Aluminum chamber-rotary stage adapter platform:

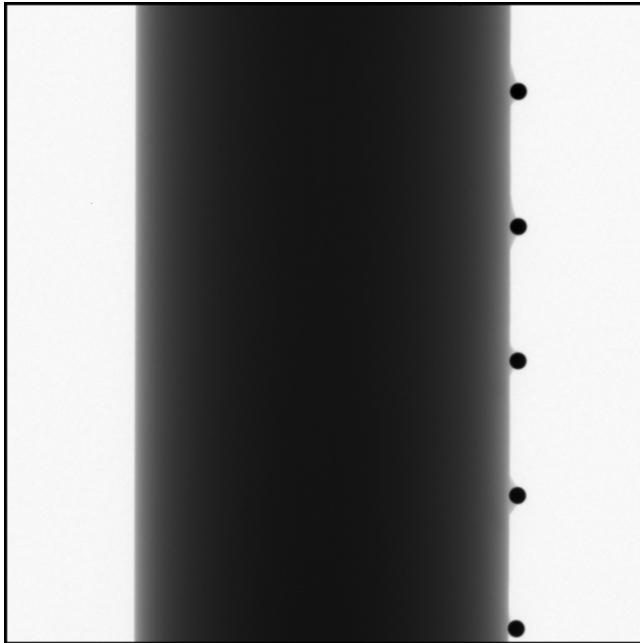


### **X-ray CT Geometric Calibration Scanner**

Following the suggestion of Dr. Andó from the University of Grenoble, an X-ray calibration device and procedure was designed to accurately extract the geometric parameters critical for a successful reconstruction. The device was constructed by attaching steel balls ( $\text{Ø} = 1/8''$ ) along the length of a white, Delrin plastic rod. The calibration cylinder is then placed tightly within the hole in the aluminum chamber-rotary stage platform to complete the calibration setup.

Next, a set of projections is taken at a set increment during a  $360^\circ$  rotation. Only 6 projections are required; however, the accuracy of the extracted parameters increases with the number of obtained projections. An even set of projections is required.

An example projection during a calibration scan.



Trajectory of two balls.



Subsequently, the projections must be processed to attain the position of the center of each ball for each degree increment. ImageJ is implemented for all image processing. The procedure is as follows:

- 1) Threshold the image to segment the steel balls from all other materials.
- 2) Select 2 balls that make complete trajectories through all projections and exist on opposite sides of the vertical midpoint
- 3) Subtract the remaining balls from the projections.
- 4) Obtain the center of the balls in each image by calculating the center of mass

With a complete set of coordinates for each ball in each image, the algorithm, written in Matlab, can be called to process the data. The geometric calibration algorithm is a simplified approach from Noo et al. (2000). The algorithm presumes that balls define an elliptical orbit when the projections are stacked.

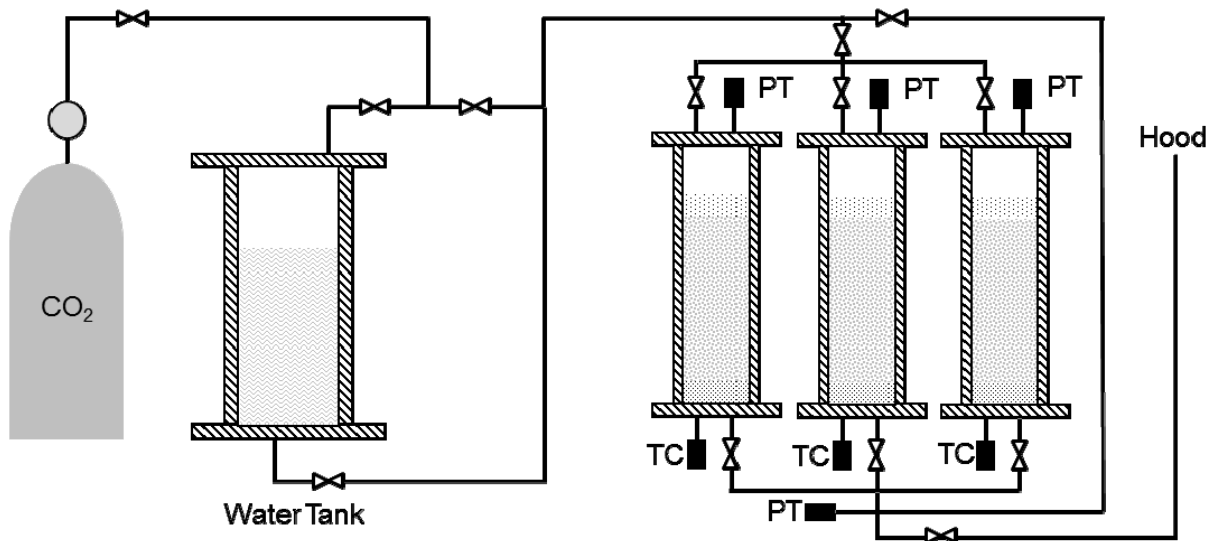
The first objective is to calculate the axis tilt, which most reconstruction algorithms require. This is calculated by determining the angle by which the line passing through the projected centers of

each ball's orbit differs from the vertical axis. The projection of the centers of the orbit is calculated by performing a least squares fit for the point at which each line connecting balls from projections 180° apart intersect. The coordinates are then corrected by rotating them by the tilt angle.

Next, a least squares analysis is performed to fit ellipses to the balls' orbits. The equation to describe the ellipse is described by a 4th order polynomial. The source detector distance (SDD) can then be calculated by manipulating the ellipse parameters (Noo et al. 2000). Finally, the source object distance (SOD) can be estimated by an iterative approach. This calculation however requires the knowledge of the exact distance between the projected balls; consequently, inaccuracies in this distance propagate to errors in the estimated SOD.

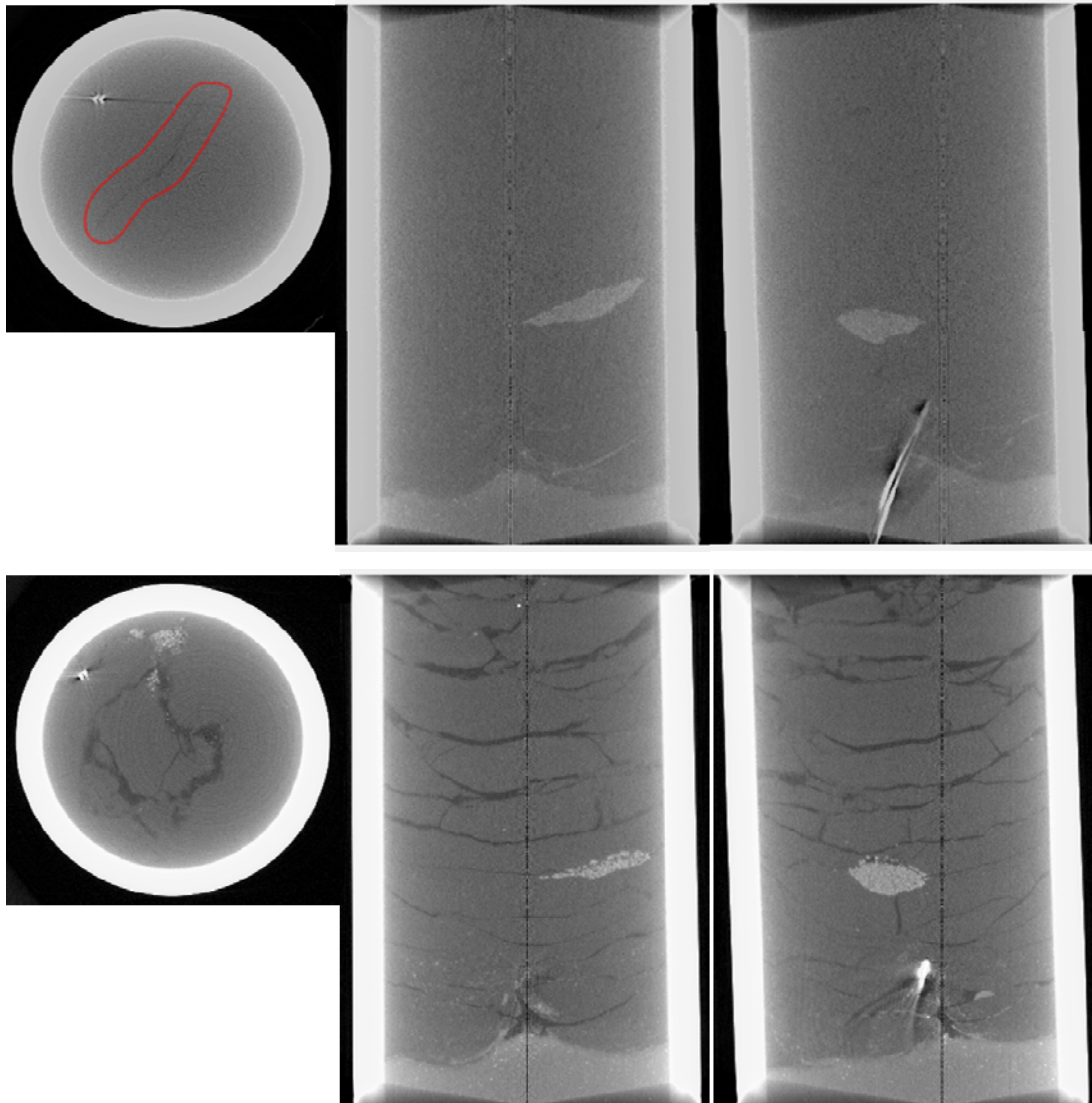
### CO<sub>2</sub> hydrate formation in various conditions

CO<sub>2</sub> hydrate was formed in different conditions within the X-ray transparent high-pressure sediment chambers. The schematic demonstrates the ability of the setup to perform multiple hydrate formation experiments simultaneously and the flexibility of the system to allow for the injection of gas and water to both the top and bottom of the chambers.



## Strategy based on Dry Diatoms

The idea for the use of diatoms was stimulated by the recognition of the existence of diatom-rich sediments in many known locations of hydrate in fine-grained sediment (e.g. Ulleung Basin, Blake Ridge). The concept for this experiment was to use the inner space of the diatom particles as a storage space for CO<sub>2</sub> gas, and therefore, as water was injected, hydrate could easily form due to adequate close, supply of gas and would not be limited by the long time scale diffusion process. Hydrate was formed as evidenced by P-T trajectories during formation and dissociation le gas driven fractures. Image before and after dissociation follow.





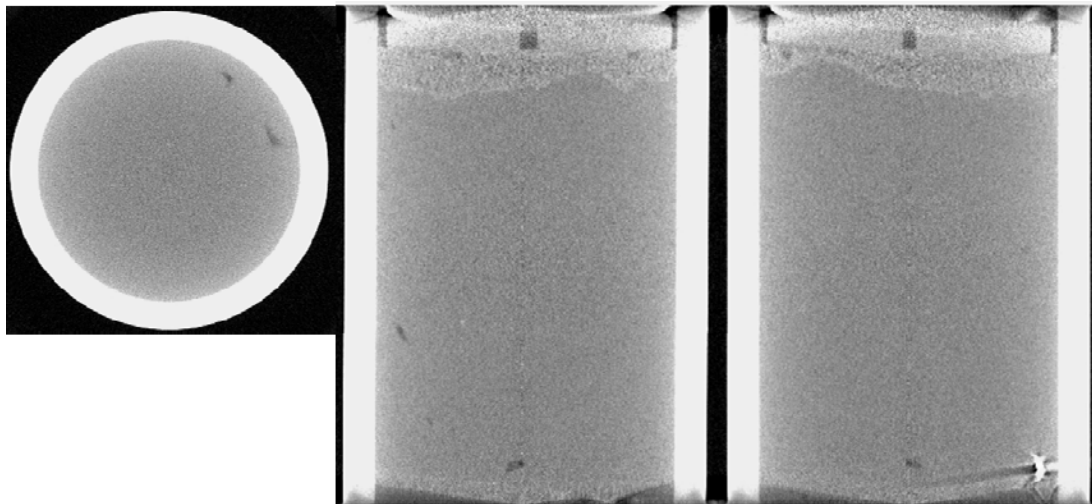
### **Strategy based on Frozen Dry Kaolinite with Ice Lens**

For this condition, the idea was to transform an ice lens placed in dry kaolinite directly to hydrate with a sufficient supply of CO<sub>2</sub> gas. The sediment was then injected afterwards with a CO<sub>2</sub> saturated solution to further the hydrate formation. No direct ice-to-hydrate transformation was observed in the experiment (suction competes with hydrate formation). Yet, the PT trajectory during depressurization indicates that hydrate was present in the sample.

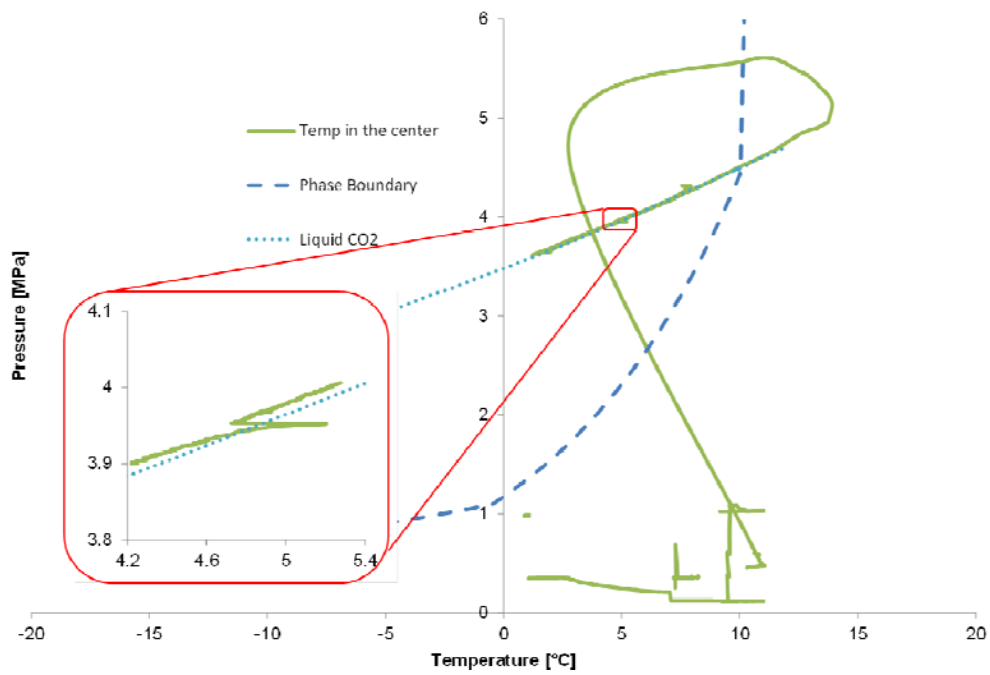
### **Strategy based on gas injection**

The idea behind this condition was to inject CO<sub>2</sub> into the water saturated sediment to cause a gas-driven fracture where hydrate would nucleate. The following sequence of images document the evolution of the test

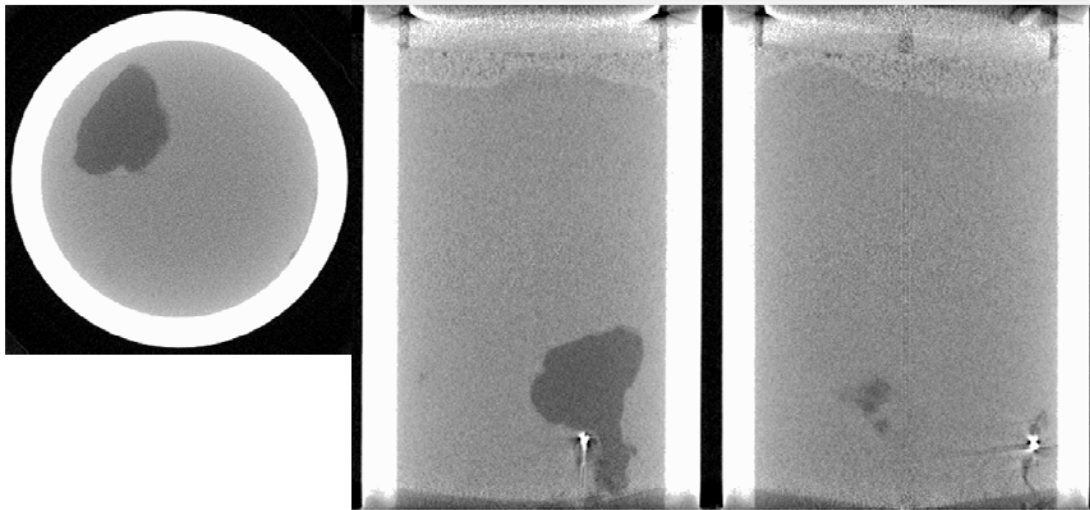
Initial condition:



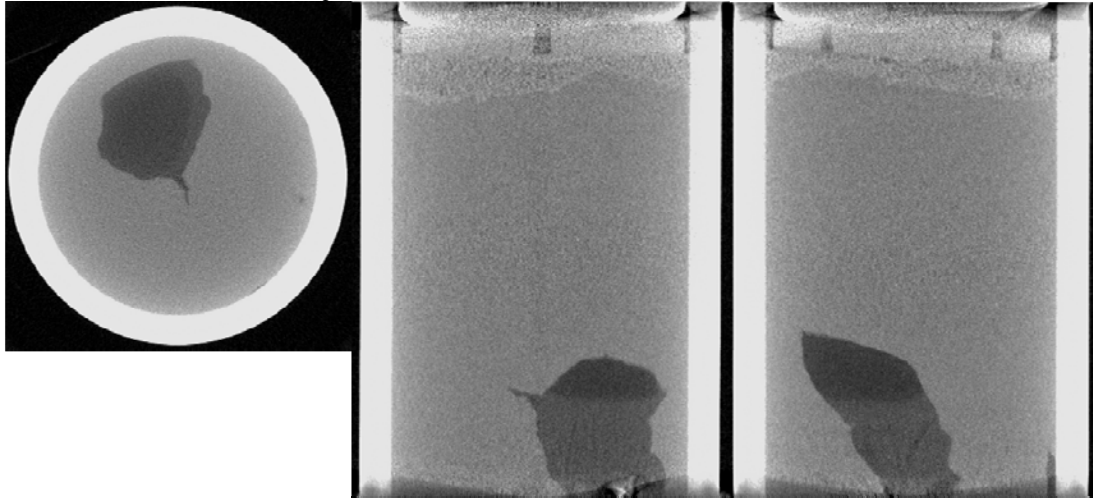
The PT trajectory when high pressure CO<sub>2</sub> is injected into the sediment:



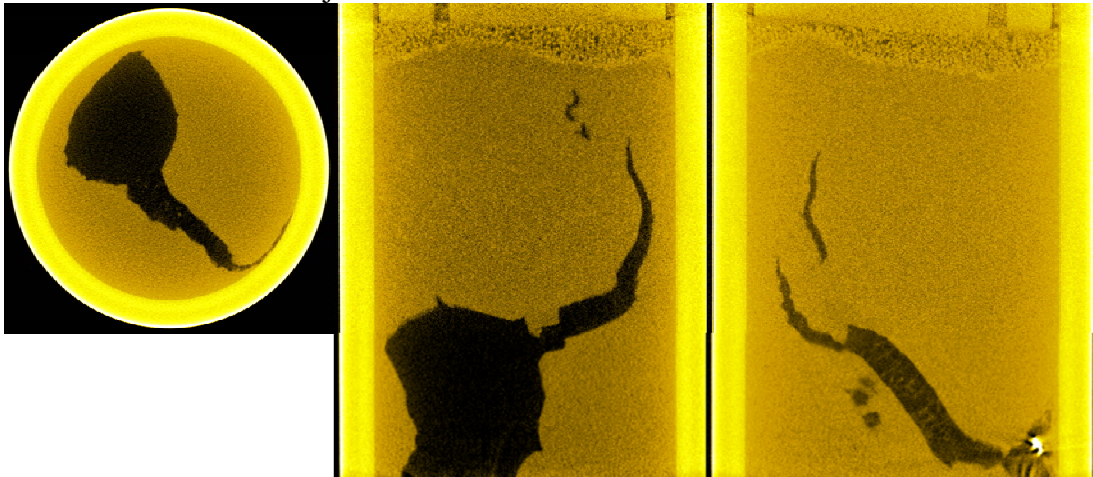
24 hours after the CO<sub>2</sub> injection:



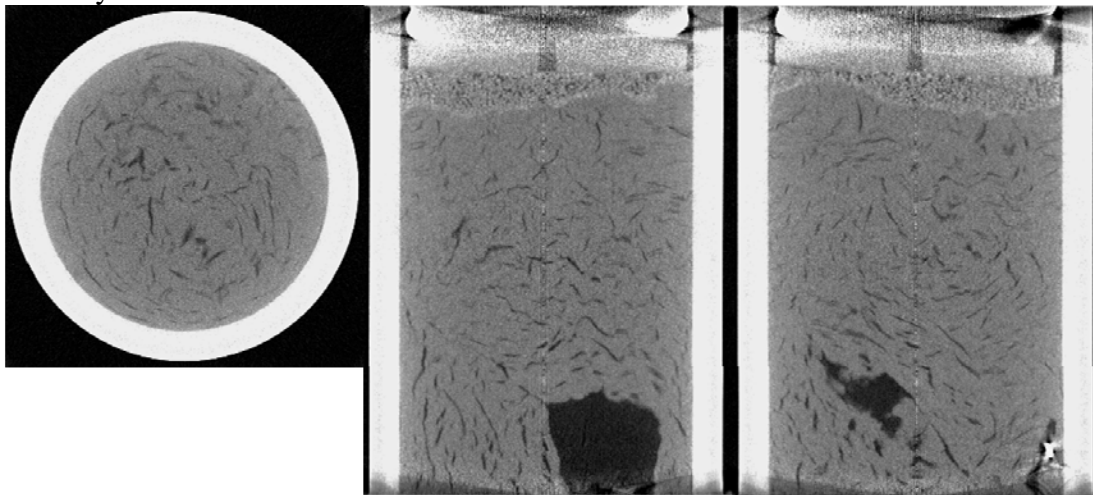
48 hours after the CO<sub>2</sub> injection:



96 hours after the CO<sub>2</sub> injection:



After hydrate dissociation:



## MILESTONE LOG

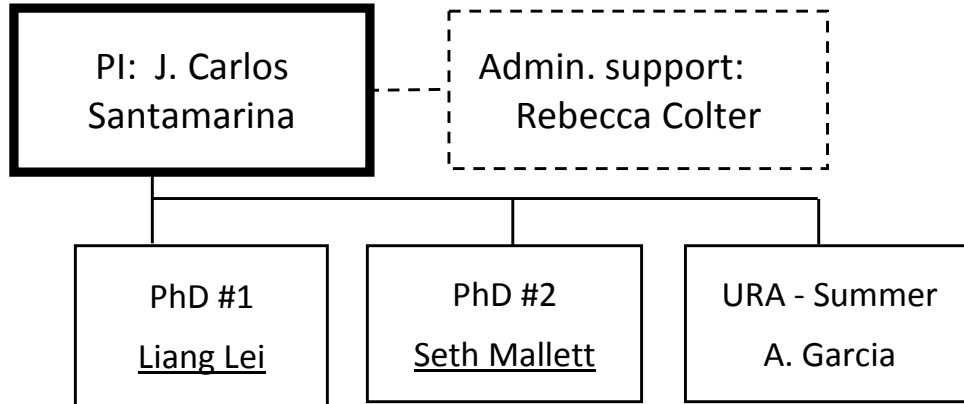
Milestone	Planned completion date	Actual completion date	Verification method	Comments
Literature review	5/2013	5/2013	Report	Completed first phase. Will continue throughout the project
Preliminary laboratory protocol	8/2013	8/2013	Report (with preliminary validation data)	this and previous reports
Cells for Micro-CT	8/2013	8/2013	Report (with first images)	this and previous reports
Compilation of CT images: segregated hydrate in clayey sediments	8/2014	In progress	Report (with images)	
Preliminary experimental studies on gas production	12/2014		Report (with images)	
Analytical/numerical study of 2-media physical properties	5/2015	In progress	Report (with analytical and numerical data)	
Experimental studies on gas production	12/2015		Report (with data)	
Early numerical results related to gas production	5/2016	In progress	Report	
Comprehensive results (includes Implications)	9/2016		Comprehensive Report	

## PRODUCTS

- **Publications:**  
In progress
- **Presentations:**  
In progress
- **Website:** Publications and key presentations are included in <http://pmrl.ce.gatech.edu/> (for academic purposes only)
- **Technologies or techniques:** X-ray tomographer and X-ray transparent pressure vessel
- **Inventions, patent applications, and/or licenses:** None at this point.
- **Other products:** None at this point.

## **PARTICIPANTS & OTHER COLLABORATING ORGANIZATIONS**

*Research Team:* The current team is shown next. We anticipate including external collaborators as the project advances



## **IMPACT**

While it is still too early to assess impact, we can already highlight preliminary success of exploring hydrate lenses morphology in real systems, and analogue studies using a high resolution tomographer.

## **CHANGES/PROBLEMS:**

None at this point.

## **SPECIAL REPORTING REQUIREMENTS:**

We are progressing towards all goals for this project.

## **BUDGETARY INFORMATION:**

As of the end of this research period, expenditures are summarized in the following table.

Note: in our academic cycle, higher expenditures typically take place during the summer quarter.

Baseline Reporting Quarter DE-FE009897	Budget Period 1								Budget Period 2							
	Q1		Q2		Q3		Q4		Q1		Q2		Q3		Q4	
	Cumulati ve Total	10/1/12 - 12/31/12	Cumulati ve Total	1/1/13 - 3/31/13	Cumulati ve Total	4/1/13 - 6/30/13	Cumulati ve Total	7/1/13 - 9/30/13	Cumulati ve Total	10/1/13 - 12/31/13	Cumulati ve Total	1/1/14 - 3/31/14	Cumulati ve Total	4/1/14 - 6/30/14	Cumulati ve Total	7/1/14 - 9/30/14
	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4
<b>Baseline Cost Plan</b>																
Federal Share	36,664	36,664	73,327	36,664	109,991	36,664	146,654	36,664	185,232	38,578	223,811	38,578	262,389	38,578	300,967	
Non-Federal Share	10,922	10,922	21,844	10,922	32,765	10,922	43,687	10,922	54,937	11,250	66,186	11,250	77,436	11,250	88,685	
Total Planned	47,585	47,585	95,171	47,585	142,756	47,585	190,341	47,585	240,169	49,828	289,997	49,828	339,824	49,828	389,652	
<b>Actual Incurred Cost</b>																
Federal Share	0	0	16,173	16,173	20,191	36,364	66,556	102,920	22,923	125,843	16,448	142,290	89,396	231,687		
Non-Federal Share	0	0	52,426	52,426	13,106	65,532	0	65,532	28,443	93,975	28,443	122,418	-45,818	76,600		
Total Incurred Costs			68,600	68,600	33,297	101,897	66,556	168,453	51,366	219,818	44,891	264,709	43,578	308,287		
<b>Variance</b>																
Federal Share	-36,664	-36,664	-20,490	-57,154	-16,473	-73,626	-43,734	-99,368	-15,656	-59,389	-22,131	-81,520	50,818	-30,702		
Non-Federal Share	-10,922	-10,922	41,505	30,583	2,184	32,767	-10,922	21,845	17,194	39,039	17,194	56,232	-57,068	-835		
Total Variance	-47,585	-47,585	21,015	-26,571	-14,289	-40,859	-21,888	-77,523	1,538	-20,351	-4,937	-25,288	-6,250	-31,537		

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