Statement of Project Objectives

A multi-scale experimental investigation of flow properties in coarse- grained hydrate reservoirs during production

DE-FE0028967

A. OBJECTIVES

The objective of this project is to gain insight into the relative permeability behavior and depressurization response of coarse-grained methane hydrate deposits subjected to perturbation through observation of behaviors at the macro (core) scale and examination of the underlying processes controlling the behaviors at the micro (pore) scale.

B. SCOPE OF WORK

The Recipient will plan and execute experimental measurements on methane hydrate-bearing sandstones to study relative permeability and depressurization. Experiments will measure these properties at the macro-scale and the Recipient will then illuminate the underlying physics through pore scale observations with micro-CT and micro-Raman analyses. The effort will initially include building of essential equipment and demonstration of experimental capability (Phase 1) and will then move to measurements on hydrate-bearing sand packs before applying these approaches to actual intact hydrate samples (Phase 2).

C. TASKS TO BE PERFORMED

This project will be conducted in two phases of 18 months each.

PHASE 1 / BUDGET PERIOD 1

Task 1.0 - Project Management and Planning

The Recipient will work together with the DOE project officer upon award to develop a project management plan (PMP). The PMP will be submitted within 30 days of the award. The DOE project officer will have 20 calendar days from receipt of the PMP to review and provide comments to the Recipient. Within 15 calendar days after receipt of the DOE's comments, the Recipient will submit a final PMP to the DOE project officer for review and approval.

The Recipient will review, update, and amend the PMP (as requested by the DOE project officer) at key points in the project, notably at each go/no-go decision point and upon schedule variances of more than 3 months and cost variances of more than 10%, which require amendments to the agreement and constitutes a re-base lining of the project.

The PMP will define the approach to management of the project and include information relative to project risk, timelines, milestones, funding and cost plans, and decision-point success criteria.

The Recipient will execute the project in accordance with the approved PMP covering the entire project period. The Recipient will manage and control project activities in accordance with their established processes and procedures to ensure subtasks and tasks are completed within schedule and budget constraints defined by the PMP. This includes tracking and reporting progress and project risks to DOE and other stakeholders.

Task 2.0 – Macro-Scale: Relative Permeability of Methane Hydrate Sand Packs

The Recipient will measure the permeability of gas and brine for flow of a single phase within fine grained-sands containing hydrate at hydrate saturations ranging from 0 to 50%. To achieve this, the Recipient will generate samples with controlled hydrate saturations.

Sub-Task 2.1 - Laboratory Creation of Sand-Pack Samples at Varying Hydrate Levels

The Recipient will create controllable hydrate saturation in sands. A uniform gas saturation will be created by gas flooding a 2" diameter, 24" long core packed with fine-grained sand (a 'synthetic' sample) with a known water saturation and pore water salinity, and the sample will be brought to hydrate stability conditions.

Hydrate saturations will be altered through both initial water saturation and brine salinity.

Sub-Task 2.2 - Steady-State Permeability of Gas and Water of Sand-Pack Hydrate Samples

The Recipient will measure single-phase permeability of gas and water at hydrate saturations up to what is expected from natural hydrate-bearing sand sediment cores in the Gulf of Mexico as best as can be synthesized in synthetic hydrate bearing sediment samples. The recipient is confident that a saturation of at least 50% can be achieved but higher saturations will be attempted. The recipient will consult analyses such as Boswell et al. (2012) to determine the expected range. These will be with only one mobile phase, gas or water, to compare to previous published measurements. Permeability to gas will be measured by applying a known gas flow rate, while permeability to water will be measured by injecting brine composition at 3-phase equilibrium. Measurements will be done with different hydrate saturations and in samples with different pore sizes/pore size distributions, that are created by using different sand sizes (30/40 through 100/120 sieve sizes) and distributions (fraction of different sieve sizes), and as best possible reflect distributions expected in hydrate-bearing sand sediments in the Gulf of Mexico as determined by analog data collected in shallow sand units in the northern Gulf of Mexico by Stow et al. (1986). At least 5 such samples will be prepared. Fluid saturations will be measured through effluent mass balance and/or tracers.

Task 3.0- Macro-Scale: Depressurization of Methane Hydrate Sand Packs

The Recipient will perform depressurization to simulate a production tests on sand-pack samples at a range of initial hydrate saturations from 0 to 50% or higher as described in Task 2.2 and as determined by mass-balance calculations. Pressure will be sequentially reduced to atmospheric pressure while monitoring mass and composition of expelled gas and water. Depressurization will be performed on a range of sand-pack samples with different initial gas and hydrate saturations from 0 to 50% determined by mass-balance calculations. For a subset of 3 of the experiments, the depressurization will be imaged in a medical CT device.

Sub-Task 3.1 - Depressurization Tests

The Recipient will perform depressurization on sand-pack samples at a range of initial hydrate saturations from 0 to 50% or higher as described in Task 2.2. Drawdown will be performed at a range of timescales from several hours to several weeks while simultaneously monitoring the volume of water and gas expelled.

Sub-Task 3.2 - Depressurization Tests with CAT scan

For a subset of three tests in sub-task 3.1, the tests will be imaged with X-ray CT throughout the depressurization.

Task 4.0 – Micro-Scale: CT Observation of Methane Hydrate Sand Packs

The Recipient will perform in situ micro-CT to understand the petrophysical properties of hydrate-bearing sediments at static conditions and under perturbation. These characterizations will be used to inform the macro-scale perturbation model.

Subtask 4.1 - Design and Build a Micro-CT compatible Pressure Vessel

The Recipient will design and build an X-ray transparent micro-consolidation device for 2 to 10 mm diameter (and a length of ½ to 2 times the diameter) samples capable of allowing visualization of methane hydrate systems as a function of changing pressure up to 20 MPa, temperature down to 4C, and composition that mimics field production environments. Axial effective stress will be provided by a spring with induced lateral stress from reaction of rigid walls of the vessel

Subtask 4.2 - Micro-Scale CT Observations and Analysis

The Recipient will image, at the pore scale, the hydrate system at a range of hydrate saturations reflecting the experiments done at the Macro-Scale. The Recipient will demonstrate the capability to identify pore habit and phase saturations for a sand-pack samples with a known hydrate saturation (expected range of 0 to 50%, or higher as described in Task 2.2).Ten excess gas and five dissolved gas flow experiments will be performed.

Task 5.0 – Micro-Scale: Raman Observation of Methane-Gas-Water Systems

The Recipient will perform in situ micro-Raman spectroscopy to characterize phases, chemistry and salinity, and diffusion kinetics in synthetic methane hydrate bearing sediment samples at pressure-temperature-composition conditions relevant to field production environments (P: up to 25 MPa; T: down to 4 C; X: methane hydrate phases, brine/water, and sands with nano-pores). Two specific types of measurements will be conducted: (1). 2D-3D Raman mapping of statically equilibrated samples at a given pressure-temperature conditions; (2). Raman point scans of samples during decompression (dissociation) and compression (formation) as a function of time at each given pressure-temperature conditions. These characterizations will be used to inform the macro-scale perturbation model

Sub-Task 5.1 - Design and Build a Micro-Raman compatible Pressure Vessel

The Recipient will build a pressure vessel with characteristics required for micro-Raman analysis of methane hydrate bearing sediment systems as a function of changing pressure, temperature, and composition that mimic field production environments.

Sub-Task 5.2 - Micro-scale petrochemistry
The Recipient will analyze the micro-Raman spectra to establish the petrochemistry of methane hydrate bearing sediment samples (including but not limited to volume ratios, cage occupancies and gas concentrations, morphology, salinity) at pressure-temperature-composition conditions and time scale relevant to field production environments.

Sub-Task 5.2 - Diffusion kinetics of methane release
The Recipient will generate a model of the data to extract the diffusion coefficients of gas molecules in the host phase in order to determine how molecular species diffuse in the system as the hydrate releases methane.

<u>Budget Period 2 Continuation:</u> The Recipient will not proceed beyond activities defined within Budget Period 1 / Phase 1 of the project without the formal written approval, by the Contracting Officer, of a budget period continuation application to be submitted to DOE by the Recipient. A DOE decision on the continuation of project efforts into Budget Period 2 / Phase 2 will include consideration of performance towards the objectives of the SOPO as well as measurement of performance against specific budget period success criteria and accomplishment of scheduled milestones, as defined within the Project Management Plan (Task 1).

PHASE 2 / BUDGET PERIOD 2

Task 6.0 – Macro-Scale: Relative Permeability of Methane Hydrate Sand Packs and Intact Pressure Core Samples
The Recipient will run relative permeability experiments for simultaneous flow of water and gas at a range of hydrate saturations (ranging from 0 to 50%, or higher as described in Task 2.2), hydrate positioning, and media.

Sub-Task 6.1 - Steady-State Relative Permeability Measurements of Sand-Pack Hydrate Samples
The Recipient will simultaneously measure gas/brine relative permeability at varying hydrate saturations. Uniform synthetic hydrate bearing sediment cores produced in Phase 1 will serve as the substrate; gas and brine (at three- phase salinity) will be co-injected and sectional pressure measurements will be used to calculate relative permeabilities. Long cores (1-2') will be used to avoid potential end effects. Experiments will be performed at for hydrate saturations between 0 and 50% or higher as described in Task 2.2 and hydrate positioning (pore filling or cementing). Cementing hydrate will be formed using the excess gas method while pore-filling hydrate will be formed using an annealing process through sequential warming and cooling.

Sub-Task 6.2 - Steady-State Relative Permeability Measurements of Intact Pressure Cores
The Recipient will simultaneously measure gas/brine relative permeability on intact pressure core samples acquired by GOM2 Marine Test – DOE project DE-FE0023919 (activity dependent upon availability of appropriate samples). Mini-PCATs will be used to place sample into a permeameter chamber specifically designed to receive pressure-core samples at

pressure. Gas and water will be co-injected and relative permeability will be measured.

Task 7.0 – Macro-Scale: Depressurization of Methane Hydrate Sand Packs and Intact Pressure Core Samples

The Recipient will run dissociation experiments at a range of hydrate saturations, hydrate positioning, and media. Pressure cores will likely have variable hydrate saturation that will be unknown at the time of sampling, and will be determined by gas volume and composition analyses. Pressure cores will likely have variable sediment composition that will be determined by post-depressurization particle size and mineralogical analysis.

Sub-Task 7.1 - Depressurization of sand-pack hydrate samples

The Recipient will depressurize sand-pack hydrate samples at a systematic range of hydrate saturations from 0 to 50% or higher as described in Task 2.2as a continuation of Task 3.0.

Sub-Task 7.2 - Depressurization of intact pressure cores

The Recipient will depressurize intact macro-scale samples acquired during GOM^2 Marine Test (DOE Project DE-FE002391. The Recipient will perform systematic slow depressurizations examining thermodynamic stability throughout the depressurization. This activity is dependent upon availability of pressure cores to the project.

Task 8.0 – Micro-Scale: CT experiments on Gulf of Mexico Sand Packs

The Recipient will depressurize intact micro-sale plugs from samples acquired during GOM2 Marine Test (DOE Project DE-FE002391. The Recipient will perform systematic slow depressurizations examining thermodynamic stability throughout the depressurization. Slow depressurization allows for the careful observation of the pressure at which dissociation begins, allowing for the calculation of in situ salinity, thus characterizing the thermodynamic conditions (pressure, temperature, salinity) of the sample. This activity is dependent upon availability of pressure cores to the project.

Sub-Task 8.1 - GOM^2 Sample Preparation for Micro-CT

The Recipient will prepare samples from Gulf of Mexico pressure cores obtained through GOM^2 (DOE project DE-FE0023919). These samples will include plugs from depressurized cores and from pressure cores as available. Hydrate formation at original saturation (as determined from well logs and geophysical/degassing tests on pressure cores) will be achieved on samples for depressurized cores.

Sub-Task 8.2 - Production Testing on GOM^2 Samples Observed with Micro-CT

The Recipient will perform production tests in the micro-consolidation device on samples prepared in Subtask 8.1. In these tests a hydrate-bearing sediment sample is taken outside the stability field by depressurization while pressure, temperature are monitored and water/gas/hydrate saturation and pore occupancy are observed.

Task 9.0 - Micro-Scale: Raman Observation on hydrate-bearing sand packs

The Recipient will conduct 2D and 3D micro-Raman imaging on natural methane hydrate bearing sediment samples from GOM2 at pressure, temperature and salinity conditions relevant to processes anticipated for depressurization based production of methane from hydrate bearing sediments. Analysis of the 3D micro-Raman spectra will allow identification of phases and molecules present in the samples including the interfaces between methane hydrate grains and sands in micro-submicron scale spatial resolution. The Recipient will reconstruct 3D imaging in micro resolution to illustrate the geometry, volume ratio, and methane concentration in the samples. Analysis of the Raman spectra with variations of pressure-temperature and composition on these samples as a function of time will be used to probe the kinetics of methane release and migration from the methane hydrate phase into the surrounding sand and brine medium.

This activity is dependent upon availability of pressure cores to the project. If samples are not available from GOM2, additional sediment samples will be prepared replicating grain size distribution, porosity and mineralogy from target locations in the Gulf of Mexico.

Sub-Task 9.1 - 3D Imaging of methane hydrate sandpacks

The Recipient will prepare natural methane hydrate bearing sediment samples of at least inches in size for 3D micro- Raman imaging with sub-micro spatial resolution and sub-wavenumber (cm^{-1}) spectral resolution. Since some “dirty” sand grains may obscure Raman imaging quality in depth, the Recipient will prepare samples with optical window access in three dimensions, as needed, to allow reconstruction of 3D imaging of the samples. The 3D imaging will show the geometry and volume ratios of the phases present, molecule concentrations and salinity in the system.

Sub-Task 9.2 - 2D Micro-Raman Imaging of methane hydrate sandpacks

The Recipient will perform 2D Raman imaging of natural methane hydrate bearing sediment samples from GOM2 at depressurization conditions. The Recipient will monitor the change of salinity at the interface of the methane hydrate phase and brine relevant to production of methane form natural hydrate bearing sediments. This will allow visualization of the dissociation of the methane hydrate phase as a function of pressure decreasing with time.

The Recipient will analyze time-dependent Raman data to construct the phase stability, chemistry, and kinetics of the system. The Recipient will build 3D images of the natural methane hydrate bearing sediment samples from UT Hydrate Center with micro-Raman analysis with sub-micro resolution. In-situ 2D mapping of the phases and chemistry of the natural samples at depressurization will be generated.

D. DELIVERABLES

The periodic, topical, and final reports shall be submitted in accordance with the attached "Federal Assistance Reporting Checklist" and the instructions accompanying the checklist. As shown in the Table below.

List of Project Deliverables			
Phase	***Task***	***Deliverable / Description***	
Phase 1	**1**	Project Management Plan	
	1	Data Management Plan	
	2	Report on production of sand-pack hydrate samples	These will be combined into one Phase 1 Report
	3	Report on depressurization of sand-pack hydrate samples	
	4	Report on design of the micro-consolidation device	
	5	Report on Micro-Raman Analysis	
Phase 2	**6**	Report on relative permeability measurement in both sand- pack and intact cores	These will be combined into one Phase 2 Report
	7	Report on depressurization of sand-pack and intact hydrate samples	
	8	Report on sampling from pressure cores	
	9	Report on Micro-Raman Analysis	

E. BRIEFINGS AND TECHNICAL PRESENTATIONS

The Recipient will participate in a project kick Off Meeting to be held at the NETL facility located in Pittsburgh, PA or Morgantown, WV (or at an alternative location or by alternative means, such as web- ex, if mutually agreed upon by the DOE and Recipient) within 90 days of the initiation of the agreement.

- The Recipient will prepare and present detailed briefings for the project officer at the NETL facility located in Pittsburgh, PA or Morgantown, WV (or alternate location or by alternative means, such as web-ex, if mutually agreed upon by the DOE project officer and the Recipient). Briefings will be given by the Recipient to explain the plans, progress, and results of the technical effort at least once per year.
- DOE may choose to substitute Recipient participation in external peer review or contractor review meeting for this annual briefing.
- The Recipient will prepare and present briefings at key project decision points, and/or project phase or budget period transitions. Every effort will be made to hold these meetings remotely via web-ex but there may be instances where presentation at an NETL site, or other location may be required by DOE.
- Briefings will be given by the Recipient to explain the status/progress, and results of the technical effort to that point and will clearly identify whether decision point criteria for success has been met and progress warrants continuation of efforts under the project.
- The Recipient will participate in a project closeout meeting to be held at the NETL facility

located in Pittsburgh, PA or Morgantown, WV (or at an alternative location to be agreed upon by DOE and Recipient) within the final 60 days of the agreement. The Recipient will, as part of that meeting, prepare and present a summary of project efforts, findings and conclusions in the context of planned research and project objectives.