

Heterogeneous Sediment Experiments

Materials and methods

Data were collected using a fiber optics-based Luna[®] Distributed Sensing System (DSS) that records small changes in temperature and strain every one centimeter over the length of a thin optical fiber (Figure 1). Readings were taken every minute. Due to the fiber optic technology the DSS utilizes, it cannot differentiate between changes in temperature and strain; therefore, data is recorded as a temperature and strain hybrid, referred to here as the Temperature Strain Value (TSV). The system and accompanying software are designed to take baseline information of the fibers and eliminate background effects or other influences relating to the individualities of a given fiber. The data gathered expresses only changes in TSV local to the fiber. Each fiber was coiled in a spiral and attached to a circle of plastic mesh, with the beginning of the fiber on the outside of the spiral and the end of the fiber at the center of the spiral (figure 1B); thus, the further the distance along the fiber, the closer to the center of the spiral. This setup created a two dimensional “fiber plane” over which the fiber could record TSV.

The goal of these experiments was to simulate the percolation of methane gas from the subsurface through sediments to see how and where hydrate forms under these conditions. Experiments were conducted in a 33-L PVC bucket with polycarbonate windows. The bucket was filled with alternating layers of sand (2 layers) and silt (one layer), which can be seen in figure 2. The first layer consisted of seven liters of water-saturated, sieved (<500 micron grain size) commercially available Ottawa sand, the second layer was 4 liters of water-saturated silt, and the third layer was seven liters of the Ottawa sand. Four data-gathering fiber optics planes were layered within the sediment at 10, 20, 25, and 40 centimeters height and corresponded to the middle of the lower sand layer, the top of the lower sand layer, the middle of the silt layer, and the middle of the upper sand layer. A methane injection tube was also placed within the sand. The injection tube consists of 1/16” tubing with three outlet tubes of 1/8” diameter located in the sand at the bottom of the bucket.

The bucket was placed inside Oak Ridge National Laboratory’s Seafloor Process Simulator (SPS), a cylindrical 72-L volume Hastelloy C- 22 vessel with 42 sampling ports, including 2 sapphire windows, capable of maintaining pressures up to 20 MPa (see Figure 3). The space between the bucket and the inside of the SPS was filled with chilled water. Two thermocouples recorded temperature in the headspace and the water surrounding the bucket, respectively. The SPS was housed in an explosion-proof coldroom and cooled to ~1 °C for the duration of each experiment. The optical fibers were connected to the DSS through a pass-through port in the side of the SPS. Optical fibers used ranged in length from 100 to 400 centimeters (100 to 400

sensors). For both experiments the system was set up the day before the experiment using chilled sand mixed with chilled water to 100% saturation and layered with fiber planes within the bucket. The bucket was placed within the SPS, the methane injection tube and the optical fibers were connected, the SPS was filled with water within 2 cm from the top of the vessel, and the SPS was sealed and placed within the coldroom. The SPS was flushed with nitrogen and then pressurized with methane to just below the hydrate stability field (HSF) as calculated using The Colorado School of Mines' CSMHYD hydrate prediction software. The pressurized vessel was left to stand overnight in the coldroom so that the methane could diffuse into the water.

A high-pressure liquid chromatography (HPLC) pump was used to control the rate of methane injection by slowly pumping water into methane-filled cylinders, thus pumping methane gas at an equal rate through the injection tube and into the sediment (figure 4). Methane injections lasted 10-14 hours at injection rates between one and four milliliters per minute with an average rate of 1.8 and 3.7 mL/minute for the August 2007 and September 2007 (figure 5) experiments, respectively. The total volume of injected methane was approximately 2017 and 1917 mLs at 400-600 psi for the August and September 2007 experiments, respectively. When the pressure inside the vessel was high enough to be well inside the hydrate stability field, the methane injection was halted and the vessel was left, pressurized, in the coldroom overnight. The next day, the vessel was depressurized and the coldroom was turned off to force hydrate dissociation through warming and depressurization simultaneously.

Data Collection and Processing

For each experiment temperature and pressure data of the vessel were collected using LabView software and the data were read into Excel and graphed. This data showed that the temperature of the vessel remained at ~ 1 °C for the duration of the experiments and showed the overall pressure events that describe vessel pressurization and depressurization. The DSS data was also read into Excel. The DSS software records an absolute time and a TSV reading for each sensor. From these values a matrix with the first row corresponding to the sensor (or alternatively the position) and the first column corresponding to time and each value within the body of the matrix a TSV value. For the first experiment there were 10096 rows of data (corresponding to data collected every 60 seconds for 7.04 days) and for the second experiment there were 7195 rows of data (corresponding to data collected every 60 seconds for 5.01 days). The number of sensors varied for each fiber. From examining the data across a fiber at different time steps including the starting time and finishing times and the time corresponding to 24, 48, 72, 96, 120, 144, and 168 hours if available. From these graphs bad sensors could be detected and their values were eliminated. This left 105 sensors for fiber 1, 200 sensors for fiber 2, 182 sensors for fiber 3, and 106 sensors for fiber 4. Next data were examined for select

sensors including the first and last sensors and sensors 25, 50, 75, 100, 125, 150, 175, and 200, if available. These graphs should help to identify regions indicating hydrate formation or dissociation. Additional 3-D surface plots and contour plots (shown in Figure 6) have been generated using the OriginPro software package.

Future Work

Several steps remain in the data processing:

- 1) Since the fibers were configured as spirals we would like to graph the data using polar coordinates.
- 2) More analysis of the data needs to be conducted to identify features that could indicate hydrate formation or dissociation.
- 3) Generation of publication quality graphs. Currently the 3-D surface graphs can only be exported without color.
- 4) Currently we are working to make a movie out of the data where each time step corresponds to a movie frame.

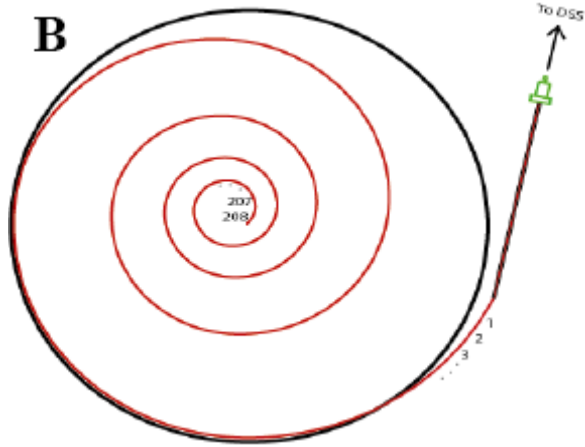
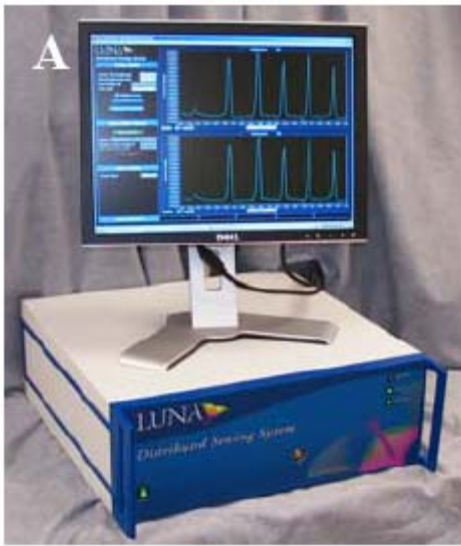


Figure 1. The Luna[®] Distributed Sensing System (A) uses laser pulses to determine changes in shape of the optical fiber due to temperature and strain changes at 1-cm intervals along the length of the fiber (B), diagrammed here in red in a spiral conformation. The laser light is reflected at each 1-cm sensor grating. Temperature and strain cause the fiber to expand, contract, and/or bend, all factors which are notable in the reflection time. The time it takes for the reflection to return to the

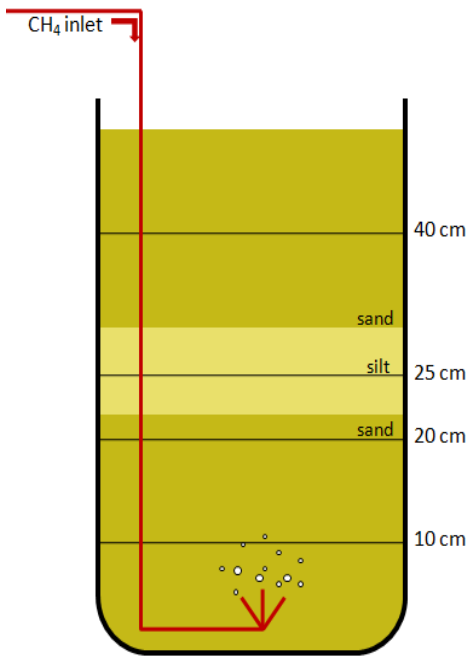


Figure 2. The 33 liter PVC bucket filled with sand, silt, DSS data planes (horizontal lines), and the methane injection tube.



Figure 3. Oak Ridge National Laboratory's Seafloor Process Simulator is a cylindrical 72-L volume Hastelloy C- 22 vessel with 42 sampling ports, including 2 sapphire windows, that is capable of maintaining pressures up to 20 MPa.

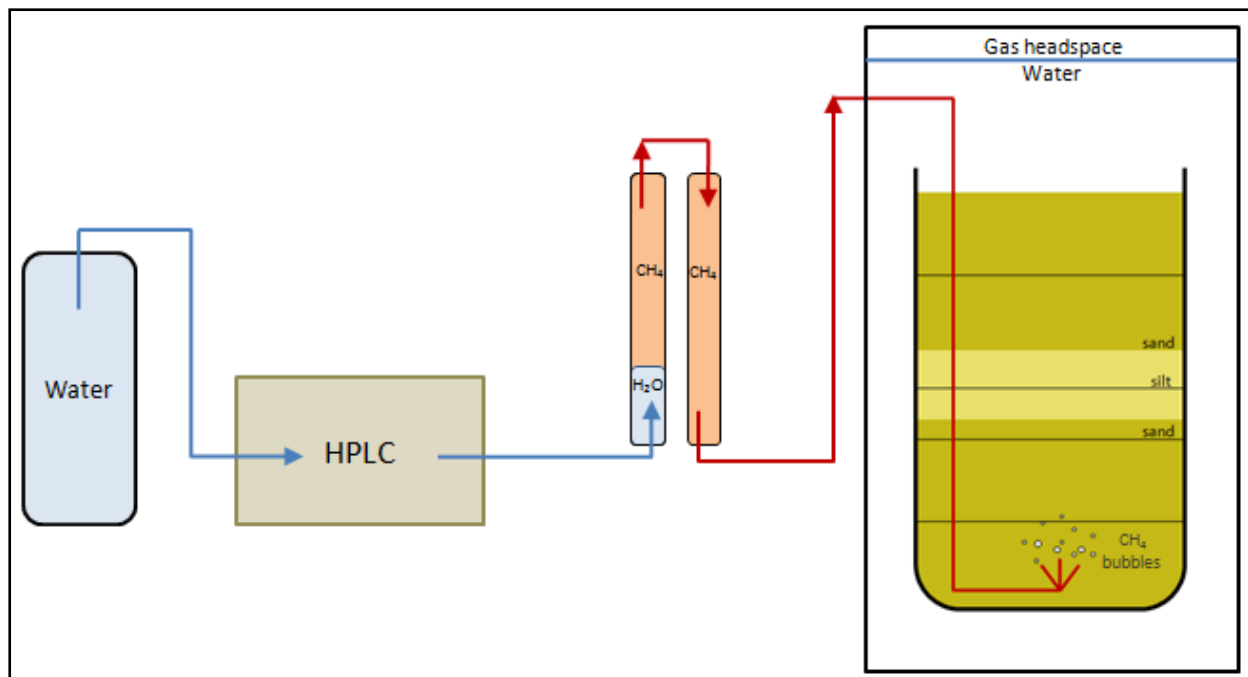


Figure 4. The methane injection system consisted of an HPLC pump which slowly pumped water into two Hoke cylinders that were pre-pressurized with methane. As the water flowed into the bottom of the Hoke cylinder, methane was displaced by an equal volume and was forced through the injection tube into the sediment.

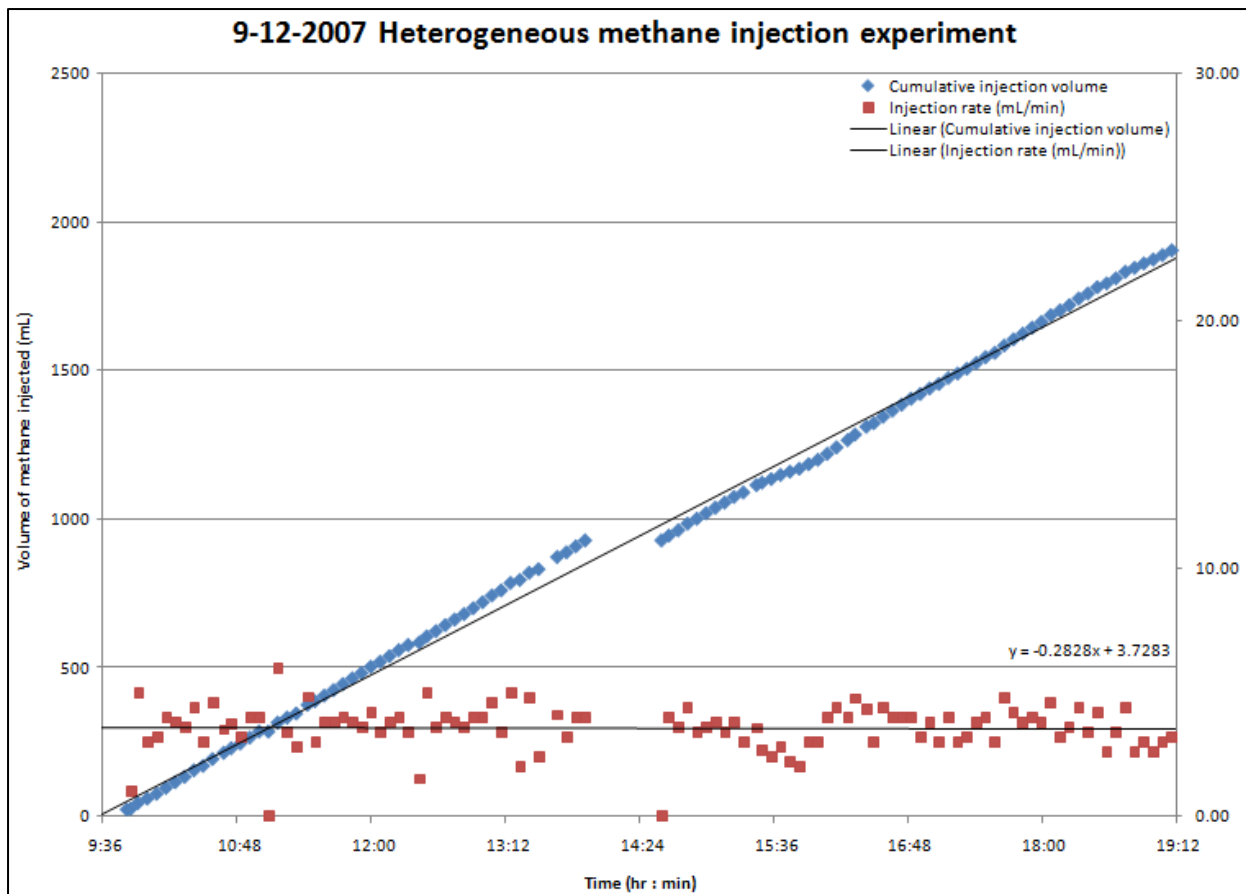


Figure 5. Cumulative injection volume and methane injection rates over the duration of the September 2007 experiment. The average methane injection rate was 3.72 mL/minute.

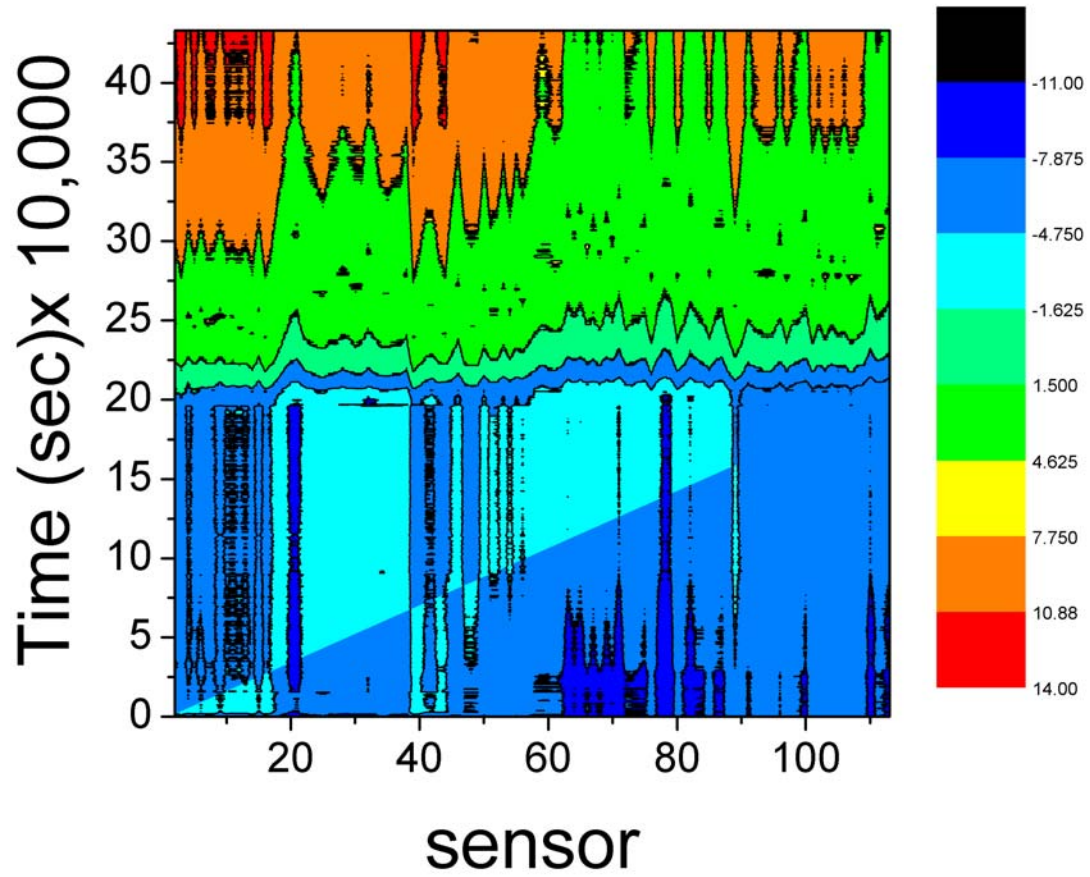


Figure 6. Contour plot from fiber 1 during Experiment 2 (September 2007).