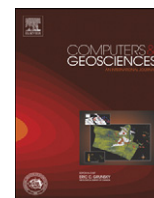




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## Interpreting temperature–strain data from mesoscale clathrate experiments

J.R. Leeman<sup>a,b</sup>, C.J. Rawn<sup>c</sup>, S. Ulrich<sup>b</sup>, M. Elwood Madden<sup>a,b</sup>, T.J. Phelps<sup>b,\*</sup><sup>a</sup> School of Geology and Geophysics, University of Oklahoma, 100 E. Boyd, Norman, OK 73019, United States<sup>b</sup> Biosciences Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, United States<sup>c</sup> Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, United States

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## ABSTRACT

Gas hydrates may play an important role in global climate change, carbon sequestration, energy production and seafloor stability. However, formation and dissociation pathways in geologically complex systems are poorly defined. We present a new approach to processing large amounts of data from a LUNA distributed sensing system (DSS) in the seafloor process simulator (SPS) at Oak Ridge National Laboratory to monitor and visualize gas hydrate formation and dissociation in heterogeneous sediments. The DSS measures relative temperature/strain change with a high spatial resolution allowing the heat of reaction during gas hydrate formation/dissociation to be used to locate clathrate processes in space and time within the vessel. Optical fibers are placed in the sediment following an Archimedean spiral design and the position of each sensor is determined iteratively over the arc length using Newton's method. The DSS data are then gridded with a natural neighbor interpolation algorithm to allow contouring. The locations of sensors on the fiber were verified with hot and cold stimuli in known locations. Software was developed to produce temperature/strain linear and polar plots, which aid in locating significant hydrate formation/dissociation events. Results from an experiment using a vertically split column of sand and silt clearly showed initial hydrate formation in the sand, followed by slow encroachment into the silt. Similar systems and data processing techniques could be used for monitoring of hydrates in natural environments or in any situation where a hybrid temperature/strain index is useful.

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## 1. Introduction

Gas hydrates, a type of clathrates, are cage-like structures of water molecules bonded to form cavities, which are populated by gas molecules such as carbon dioxide or methane. Natural gas hydrates are stable at low temperatures and moderate pressures, making the seafloor an ideal environment for methane hydrate formation. Gas hydrates are sensitive to changes in pressure and temperature; dissociation can be triggered by lowering the water column or by increasing temperature (Buffet and Archer, 2004; Makogon et al., 2007; Sloan, 1998). Hydrates are also subject to action by chemical inhibitors, which increase the required pressure and decrease the temperature conditions for clathrate stability.

Gas hydrates have been proposed as a sequestration mechanism for carbon dioxide (Brewer et al., 2000; Gabitto and Tsouris, 2006; Goel, 2006; Jadhawar et al., 2006) and hydrates of natural gases could provide significant volumes of methane for energy production (Boswell, 2007; Moridis et al., 2009; Walsh et al., 2009). Gas hydrates could also be used as a transport or storage mechanism for

gases under low temperature and moderate pressure conditions (Chatti et al., 2005). In addition, dissociation of gas hydrates may have contributed to past global warming events (Kennett et al., 2000; Beauchamp, 2004; Dickens et al., 1997; MacLennan and Jones, 2006; Max et al., 1999; Padden et al., 2001; Weissert and Erba, 2004). Dissociation of hydrates present in seafloor sediments could also result in seafloor instability (Mienert et al., 2005; Kvenvolden, 1999). However, current estimates of hydrate volume vary by many orders of magnitude (Klauda and Sandler, 2005) and constraining these values would be of great aid in determining both the role of gas hydrates in global climate change and the feasibility of producing natural gas from hydrate reservoirs. Therefore, experiments examining and visualizing the formation and dissociation pathways of gas hydrates within sediments have an impact on the assessment of both geological and industrial hazards.

High-resolution data collected through long experiments and effective visualization methods are required to understand the complex behavior of a sediment–hydrate system. Previous workers have used X-ray (Kneafsey et al., 2007) and acoustical (Waite et al., 2004) tomography, neutron diffraction (Thompson et al., 2006), and magnetic resonance imaging (Gao et al., 2005) to observe hydrate formation and dissociation in situ. Fiber optic sensing can also be utilized for effective visualization of hydrate

\* Corresponding author.

E-mail address: [phelpstj@ornl.gov](mailto:phelpstj@ornl.gov) (T.J. Phelps).

formation within sediment. This paper focuses on data processing methods developed to analyze and visualize large data sets collected by a fiber optic distributed sensing systems (DSS) within complex, multicomponent hydrate experiments in the Seafloor Process Simulator (SPS) at ORNL (Phelps et al., 2001; Rawn et al., 2011).

## 2. Materials and methods

Mesoscale laboratory experiments help bridge the gap between expensive and difficult field-based analysis of natural hydrates in situ or within preserved cores, and microscopic or molecular-scale measurements of synthetic hydrates, which lack sufficient scale to realize the complexity of natural systems. The SPS is a 72-L pressure vessel 0.33 m in diameter and 0.9 m in length. Designed and constructed at Oak Ridge National Laboratory, the SPS is capable of maintaining pressures up to 20 MPa between 271 and 288 K (Fig. 1) (Phelps et al., 2001). The SPS is made of Hastelloy, a corrosion-resistant nickel-rich steel alloy, and is equipped with 41 pass-through ports for instrumentation, visualization (sapphire windows), or fluid delivery. The vessel is protected from overpressurization by a burst disk. A cold room equipped with an explosion-proof gas evacuation system and monitoring equipment houses the SPS during experiments.

To investigate the effect of sediment size on hydrate formation pathways, a column of sediment split vertically with fine mesh to divide Ottawa sand (500  $\mu\text{m}$ ) from silt (67  $\mu\text{m}$ ) was placed within the SPS for hydrate formation experiments (C in Fig. 1).

To maintain thermal and pressure equilibrium between the heterogeneous sediment system and the gas to be injected, a

second, much smaller, 12-L pressure vessel (0.69-MPa pressure rating) containing the hydrate-forming gas (B in Fig. 1) sits on top of a 20-L sediment column within the SPS. A high-performance liquid chromatography (HPLC) pump injects a water mixture containing 2% methanol to displace gas from the secondary vessel through a branched diffuser (pressure is applied to both outlets, sand and silt) and raise the pressure of the SPS (Fig. 1). Methanol is a hydrate inhibitor and thus prevents hydrate formation inside the secondary vessel. The gas from the secondary vessel is forced through a diffuser into the lower portion of the sediment column to simulate natural gas flow in seafloor sediments. Gas is delivered simultaneously to both the sand and silt sides of the column through screened outlets connected with a 'T' fitting.

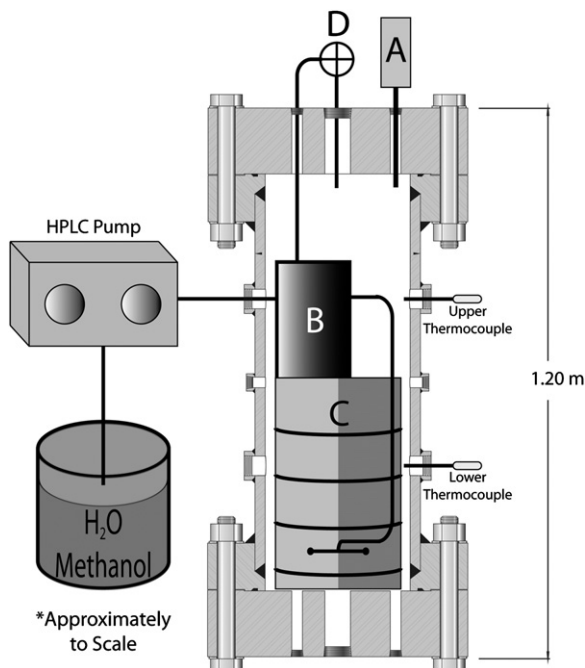
The vessel is monitored by multiple systems that record the pressure and temperature in the headspace and temperature-strain values from the DSS in the sediment throughout the experiment. Gas hydrate formation is an exothermic reaction and dissociation is endothermic. Assuming the vessel and sediment are in thermal equilibrium prior to injection, increasing temperatures within the sediment indicate areas where clathrate is likely forming and lower temperature values indicate that hydrate is likely decomposing. A LabView interface monitors the pressure and temperature of the vessel and the temperature of the cold room to determine external controls on the thermal state of the system. A LUNA Innovations distributed sensing system (DSS) monitors the temperature-strain value along fiber optic cables, which are embedded within the sediment column prior to the experiments. The fibers, coated with Teflon to help reduce the strain response in the measurements and increase their durability, are tied to a plastic mesh along a path approximated by an Archimedean spiral to provide a suitable mechanism for mathematical modeling and then embedded in the sediment. See Rawn et al. (2011) for further details on the operation of the DSS within the vessel.

The DSS measures hybrid temperature-strain values at discrete locations along the fiber optic cable. Bragg gratings are embedded at 1-cm intervals along each fiber utilized by the DSS. The optical properties of the gratings change with temperature and strain due to changes in the grating size, therefore changing the Bragg wavelength (Hill and Meltz, 1997). By sensing changes in the properties of the Bragg gratings, the DSS determines temperature-strain changes at each point.

The DSS relies upon optical principles described by Jones calculus. As light of some arbitrary initial polarization passes through an optical device, its properties change predictably. Polarization of the light can be described with a Jones vector, and the optical device it passes through can be represented by a  $2 \times 2$  Jones matrix (Jones, 1941). Approximately 100–250 gratings per fiber are read individually along each fiber 1–2.5 m long by an optical backscatter reflectometer (OBR) as it sweeps through a range of wavelengths. The returned wavelength is then interpreted as a hybrid temperature-strain value (TSV), resolving changes as small as 1  $\mu\text{strain}$  (dimensionless strain quantity, e.g.,  $10^{-6}$  m/m) or 0.1 K.

Each DSS fiber was calibrated to correct for birefringence (double refraction) and other optical effects that result from asymmetries in the fiber prior to the experiment. During and following gas injection, data are recorded from each grating in a delimited text file every minute. The system collects approximately 96,240 data points per hour, so the average week-long experiment produces approximately 16 million data points.

The  $P$ - $T$  data system produces a modest number of data in comparison to the DSS. The bulk vessel pressure is recorded to a precision of 68.9 Pa (0.1 psi), and temperature is monitored at two locations (upper and lower positions) in the vessel with a resolution of 0.1 K every minute.



**Fig. 1.** Configuration of the Seafloor Process Simulator. Two Hastelloy-cased type K thermocouples (both in free gas) and a pressure transducer (A) are used in addition to the DSS system to monitor hydrate formation and dissociation. The 12-L internal pressure vessel (B) rests on top of the sediment column to allow methane gas to equilibrate with the vessel conditions prior to injection. DSS grids are placed at 5, 15, 25, and 35 cm from the bottom of the sediment, represented as black lines in the sediment column (C). The dots at 10 cm indicate the location of the diffuser screens, which were connected via a 'T' fitting. The sediment column is vertically split with a fine mesh between sand and silt. An equilibrium control value (D) is situated outside the vessel for easy access.

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