

Oil & Natural Gas Technology

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Quarterly Research Performance Progress Report (Period ending 03/31/2014)

**Measurement and Interpretation of Seismic Velocities and Attenuations
in Hydrate-Bearing Sediments**

Project Period (10/1/2012 to 9/30/2015)

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Office of Fossil Energy

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Abstract:

Measurement and Interpretation of Seismic Velocities and Attenuations
in Hydrate-Bearing Sediments

Grant/Cooperative Agreement DE-FE 0009963.

This project to measure the seismic velocity and attenuation in methane hydrate-bearing sediments continues to make good progress. During this last period we focused on we have continued to test and develop our equipment, calculated stability fields for our experiments, and measured ultrasonic velocities and attenuations in hydrates and hydrate-bearing rocks.

- Example phase stability for the $\text{CH}_4 - \text{CO}_2 - \text{H}_2\text{O}$ system have been calculated to help design our experiments
- Ultrasonic waveforms for water, ice and THF hydrate were collected and spectral analysis performed to extract attenuation
- Ultrasonic velocities were collected in Bentheim sandstone containing different degrees of hydrate saturation
- The Torlon vessels have been developed to hold pressure in the NMR system

Further development of the ultrasonic attenuation calculation is underway to make the process more accurate and robust. Formation of hydrates in the Bentheim sandstone are heterogeneous. Velocities are roughly consistent with a combination of the pore-filling and envelope-cementing models.

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Executive Summary:

Our goal is to measure the seismic velocities and attenuations of methane hydrate-bearing sediments, compare these properties to the microscopic textures of the hydrates, and use the results to help in calibrating their geophysical signature. During this last period, we continued to test and develop our equipment, calculated stability fields for our experiments, and measured ultrasonic velocities and attenuations in hydrates and hydrate-bearing rocks.

To properly conduct our experiments, we need to know the stable forms of THF, methane, and carbon dioxide hydrates under laboratory conditions. Software is available in the chemical engineering department to perform the calculation. Example phase stability for the $\text{CH}_4 - \text{CO}_2 - \text{H}_2\text{O}$ system have been calculated. The initial experimental pressure and temperature path has been planned to create both methane and carbon dioxide hydrates.

Modeling requires knowledge of the properties of the pure hydrate components. Bulk samples of liquid water, solid water (ice) and THF hydrate were prepared in a special test cell. Ultrasonic waveforms for water, ice and THF hydrate were collected and spectral analysis performed. Quantitative attenuation is still difficult to extract, but the hydrate phase shows the spectral shift indicative of higher attenuation.

The Bentheim sandstone is a well-known quartz-rich standard material used by many organizations as a standard rock sample. We have used this sandstone in conjunction with the CT scanner to characterize the development of hydrates in this material. Both compressional and shear velocities were collected with hydrate saturations of 60% and 80%. The resulting velocities fall between the pore-filling and envelope-cementing hydrate distributions.

The Torlon vessel can be used at pressure in the NMR measurement system. Similar vessels are being developed for the CT scanner. System cooling and pressurization techniques are being developed that will not interfere with the imaging.

Accomplishments

A. Phase Behavior $\text{CH}_4 - \text{CO}_2 - \text{H}_2\text{O}$ system

Thermodynamic modelling provides us with the equilibrium phases at various pressures and temperatures. As is well known, in a water-methane system, will be stable at low temperatures and high pressures. In the more complex $\text{CH}_4 - \text{CO}_2 - \text{H}_2\text{O}$ system, the stable region for hydrates are modified as well as the composition. Figure 1 shows the stability regions for mixtures of methane and carbon dioxide. Very small amounts of carbon dioxide (0.5 mole %) will have very little influence.

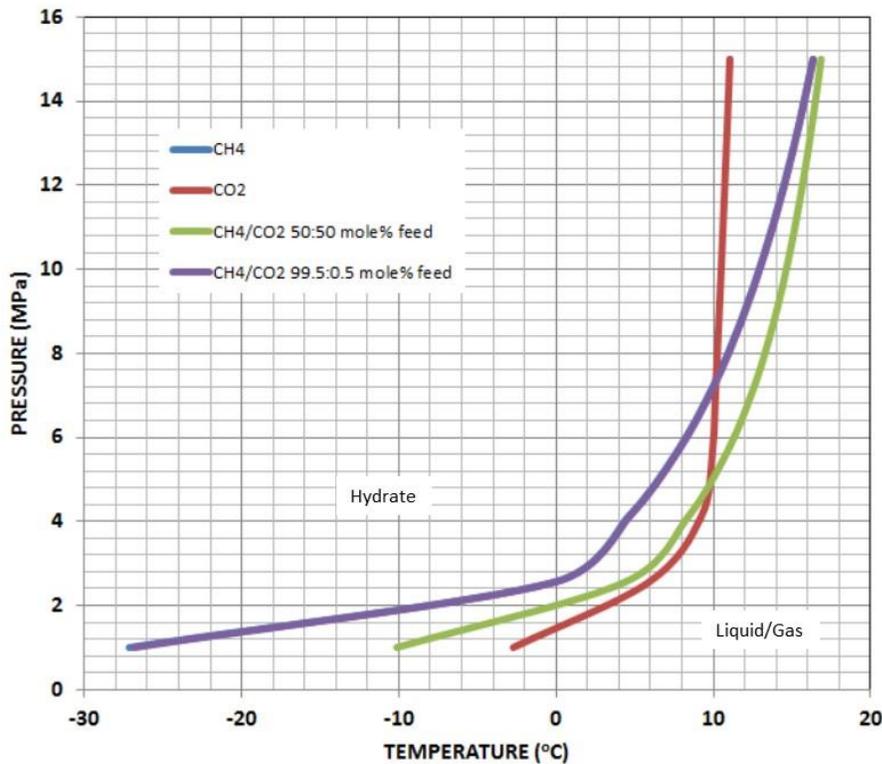


Figure 1: Extended phase relationships for the $\text{CH}_4 - \text{CO}_2 - \text{H}_2\text{O}$ system: Blue: $\text{CH}_4 - \text{H}_2\text{O}$, Red: $\text{CO}_2 - \text{H}_2\text{O}$, Green: 50-50 mixture $\text{CH}_4 - \text{CO}_2$, Purple: CH_4 with small amount of CO_2 , (note that the blue and purple lines almost coincide).

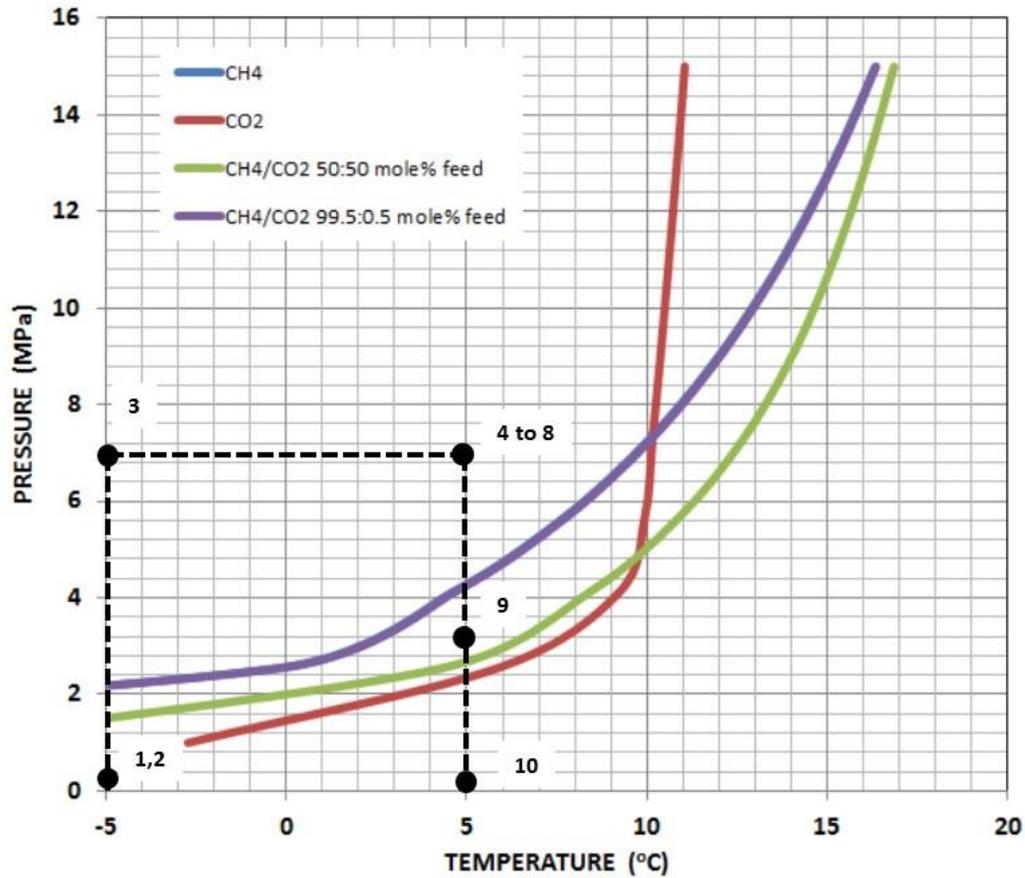


Figure 2. Similar calculations as in Fig. 1 but expanded in the near-room temperature region. Dashed line and numbers refer to a proposed experimental path (see text).

These phase calculations allow us to design our various experimental strategies. As an example of a planned procedure:

1. Crushed ice cooled in vessel to -5°C
2. Vessel vacuumed to remove air
3. Vessel pressure raised to 6.9 MPa (1000 psi) with CH_4
4. Temperature raised to $+5^{\circ}\text{C}$
5. Sample resides for several hours (CH_4 intake monitored)
6. Sample taken to test hydrate transformation
7. Methane evacuated from vessel
8. CO_2 flooded into vessel at about 3 MPa
9. Gas periodically tested for composition
10. Sample extracted and tested for conversion to CO_2 hydrate

As indicated by this proposed procedure, phase equilibrium pressure and temperature does not guarantee the formation of a particular phase. Agitation may be needed to speed conversions. Alternatively, NMR measurements are appropriate to ascertain if any free water exists in our grown hydrates. In general, we will need to employ several such techniques to quantify the actual phase distributions and compositions.

B. Ultrasonic Attenuation Measurements on THF-Water system:

Over the last couple of months we have been collecting ultrasonic waveforms of various liquids with the purpose to calculate the ultrasonic attenuation of pure (100 %) THF-hydrates. The results of our tests will help us understand what the attenuation is in pure hydrates and correlate those findings to observed attenuations in hydrate bearing sediments in nature. Similar measurements are planned in the lower frequency range to construct a data base of quality factors Q over a wide range of frequencies.

Sample Preparation and Experimental Procedure:

We continue to use THF (Tetrahydrofuran) as a hydrate former because it yields in a homogenous hydrate distribution within our sample holder. In addition, THF has a stoichiometric relationship with water which enabled us to predefine the resulting THF-Hydrate saturation. Because we wanted to investigate the attenuation of 100 % pure THF-hydrates, a mixture composing of 19 wt% THF and 81 wt% water were mashed up resulting at the right temperature condition in a 100 % THF-hydrate. The hydrate stability temperature is around 4 °C at atmospheric pressure which also guarantees ice free measurements while hydrates are stable.

To successfully conduct these experiments a sample holder needed to be designed to hold the transducers in place and at a constant and parallel distance to one and another. Figure 3 shows one of these sample holders. Little holes were drilled into the tubing and wires put through them so that the transducers were stopped from sliding too far in. Two sets of these sample holders were built, one with a distance between the transducers of 1 cm and another with 2 cm. After mixture of THF and water was prepared it was injected through the fluid lines into the sample holder. It was watched out that the entire inside of the sample holder was filled with this mixture and all of the air got pushed out leading to no visible air pocket. After this process the fluid lines were plugged so that the inside of the sample is a closed system. The whole sample holder was then connected to an oscilloscope and pulser for data recording and submerged into a temperature controlled cooling bath.

Measurements were done at three different temperatures. The first measurement was done at room temperature with the whole sample being in the liquid stage. The second measurement was performed at around 1 °C while hydrates were within the sample holder. And the third measurement was designed as a control measurement to check if there was residual water or THF within the sample at a temperature well below the freezing temperature of water (-10 °C).

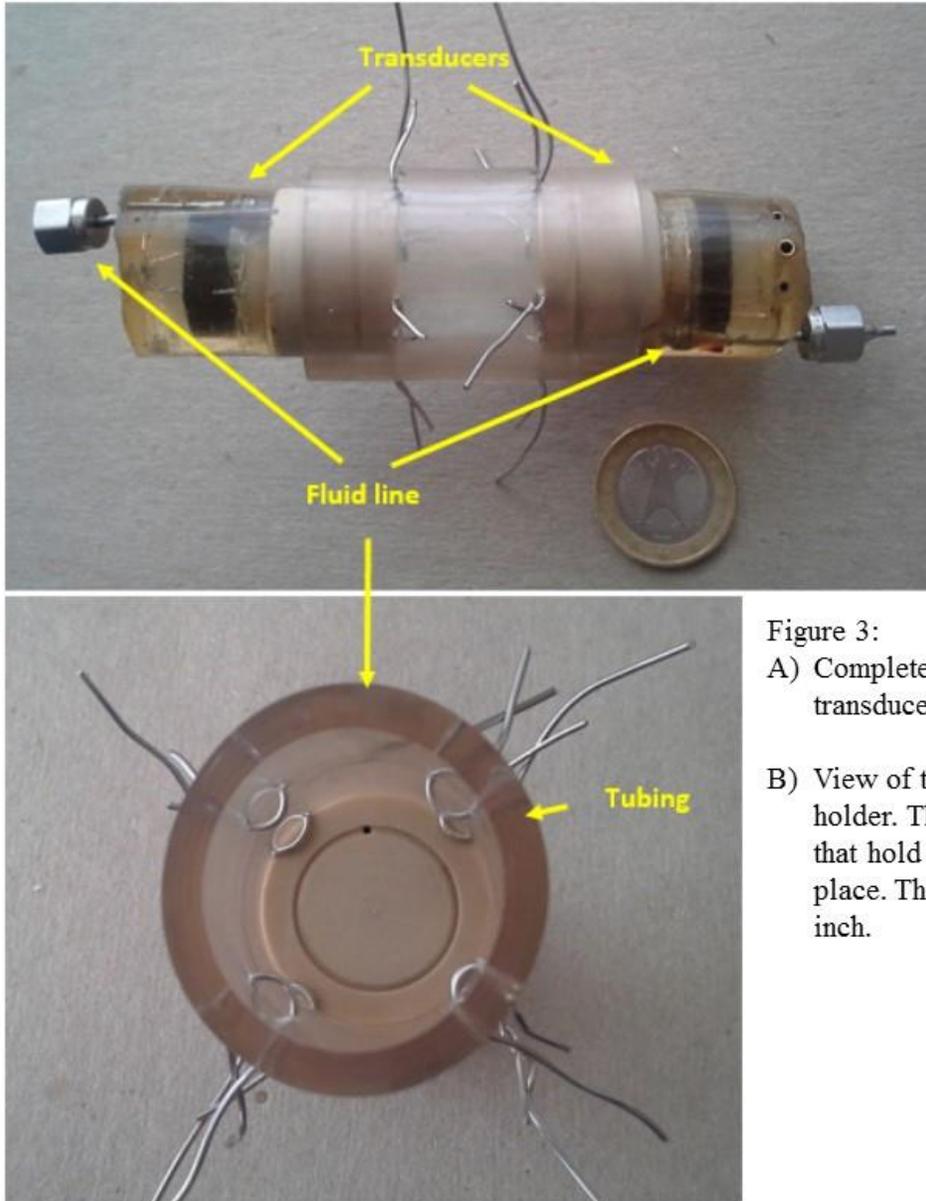


Figure 3:
A) Complete sample holder with transducers.
B) View of the inside of the sample holder. The wires can be seen that hold the transducers in place. The inside diameter is 1 inch.

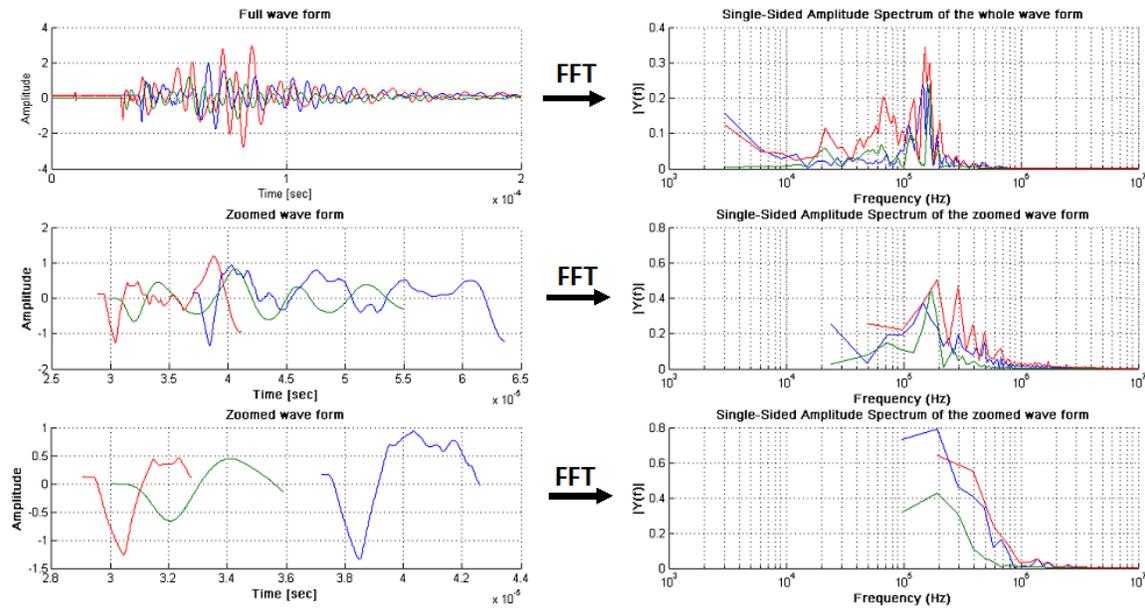


Figure 4: Collected compressional waveforms for sample at room temperature (BLUE), at 1 °C (GREEN), and at -10°C (RED) with their corresponding frequency spectrum to the right. The upper left figure shows all of the collected waveforms and to the right the whole frequency spectrum for the entire waveform. The middle figures show a zoomed in version of the collected waveform (mainly the first arrivals) and to the right their frequency spectrum. On the bottom left are only the first arrivals of the collected waveforms illustrated and to the right their corresponding frequency spectrum.

Data Processing

To obtain ultrasonic attenuation information, the data was evaluated by having an FFT (Fast-Fourier-Transformation) performed. We performed FFT's with different lengths of the recorded waveform. The first FFT was done for the whole waveform, the second for the first arrivals and a third one only for the first p-wave arrival. The collected waveforms and resulting frequency spectra for a 2 cm sample can be seen in Figures 4. To obtain the attenuation information out of the recorded data a reference spectrum needed to be calculated. Therefore we performed the same experiment with an aluminum standard that had the same length as our sample (≈ 2 cm). Again waveforms were recorded and their FFT spectra calculated (Figure 5).

It can be seen that the spectrum of the first p-wave arrival for the aluminum does not depend on temperature. This was expected and serves also as a quality control of our calculations. The next step was to divide the frequency spectrum of the aluminum standard by the frequency spectrum of the sample:

$$\frac{FFT(Aluminum)}{FFT(Sample)}$$

The results of this division can be seen in Figure 6. The desired information is contained in the first part of these figures in the frequency range from 100 kHz to 500 kHz. The slope of these calculated points provides us with the quality factor Q which is a constant value over this frequency range. We are still analyzing the results and modifying the acquisition and data reduction techniques.

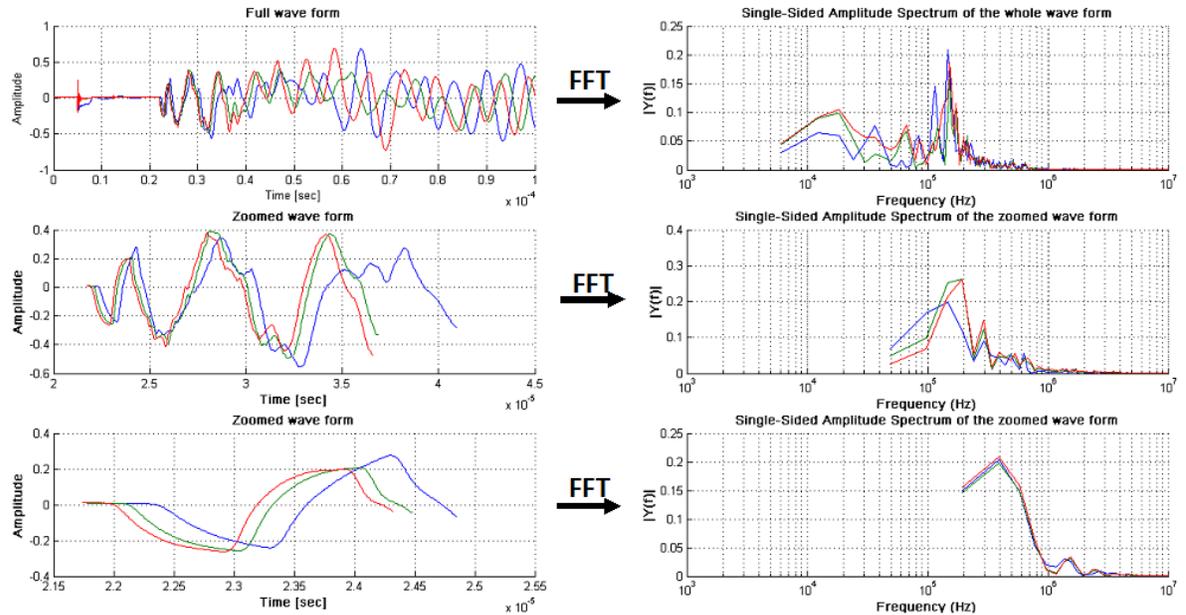


Figure 5: Collected compressional waveforms for an aluminum standard at room temperature (BLUE), at 1 °C (GREEN), and at -10°C (RED) with their corresponding frequency spectrum to the right. The upper left figure shows all of the collected waveforms and to the right the whole frequency spectrum for the entire waveform. The middle figures show a zoomed in version of the collected waveform (mainly the first arrivals) and to the right their frequency spectrum. On the bottom left are only the first arrivals of the collected waveforms illustrated and to the right their corresponding frequency spectrum.

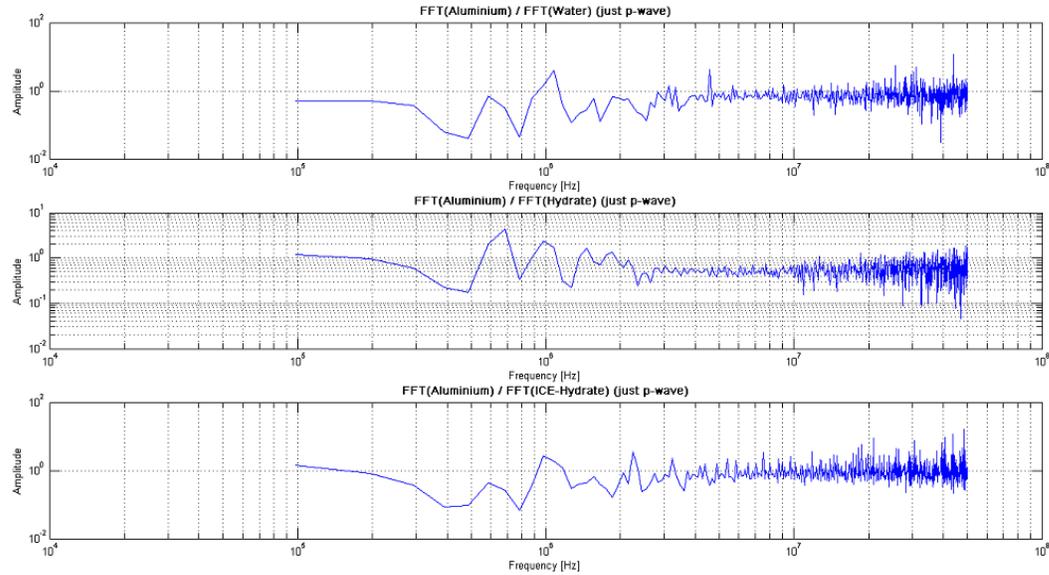


Figure 6: Spectrum divisions for the 2 cm long sample. Upper figure shows the result for the THF-water mixture, middle figure for the hydrate bearing case, and on the bottom the sample in a frozen state. All the data above 1 MHz can be considered noise and will not be considered for the attenuation calculation.

C. Hydrate formation in Bentheim Sandstone

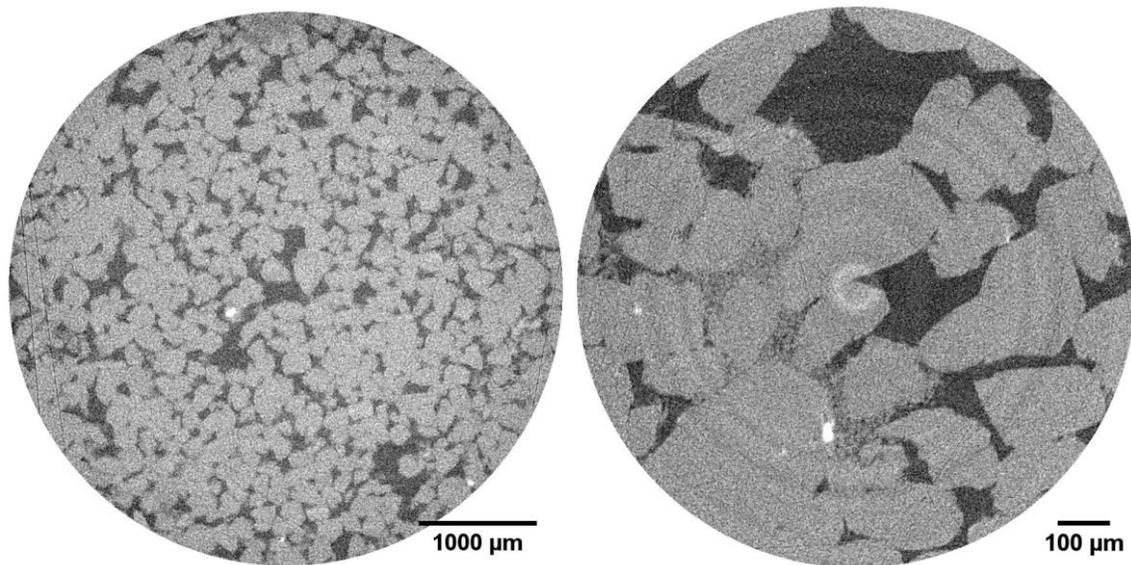


Figure 7: CT scans of Bentheim sandstone at 0.5X magnification (left) and 20X magnification (right). The clearly distinguishable grains in both images are quartz grains, the material which fills the pore space in some areas is believed to be clay (kaolinite) and the very light dots indicate grains of pyrite

Bentheim sandstone is a relatively clean sandstone (>90% quartz) which further contains feldspar (<5%) and kaolinite (<5%). It typically shows high porosities of 20-25% (Stanchits et al., 2009) and high permeabilities of 1-2 Darcy (Stevens et al., 2007). 25 ± 4 % porosity was determined from CT scans of our samples.

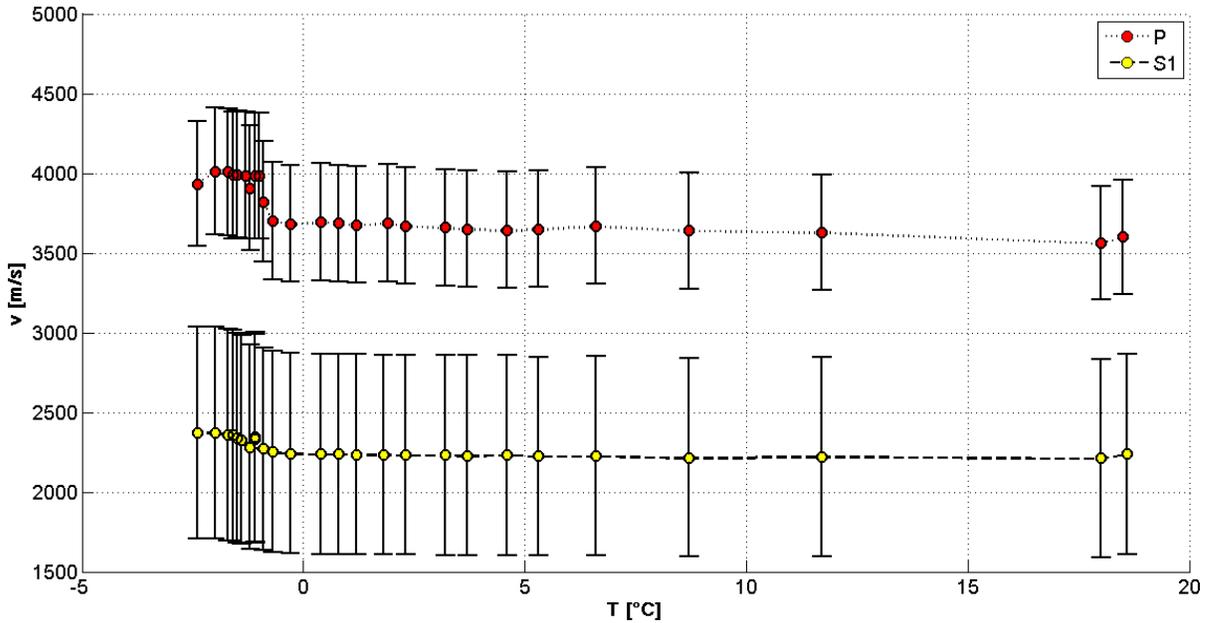


Figure 8: Ultrasonic velocities during cooling of Bentheim Sandstone sample with $S_h=60\%$

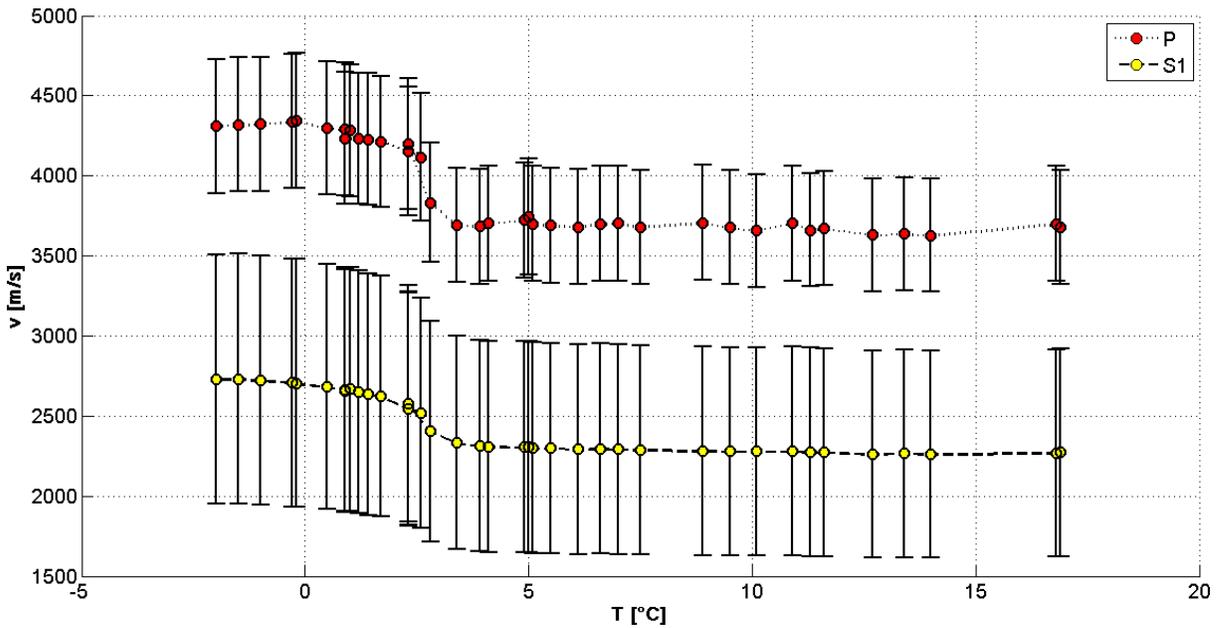


Figure 9: Ultrasonic velocities during cooling of a Bentheim Sandstone sample with $S_h=80\%$

Figures 8 and 9 show the ultrasonic P and S-wave velocities which were recorded during the cooling of Bentheim sandstone samples saturated with a water-THF mixture. Hydrate formation is indicated by an increase in velocities. Hydrates started to form around -1°C for the sample with 60% hydrate saturation and at 3°C for the sample with 80% hydrate saturation. This difference in formation temperatures is mainly attributed to differences in cooling rate.

The resulting P- and S-wave velocities before and after hydrate formation are compared to effective medium models by Ecker et al. (1998) and Helgerud et al. (1999) (Figures 10, 11 and 12).

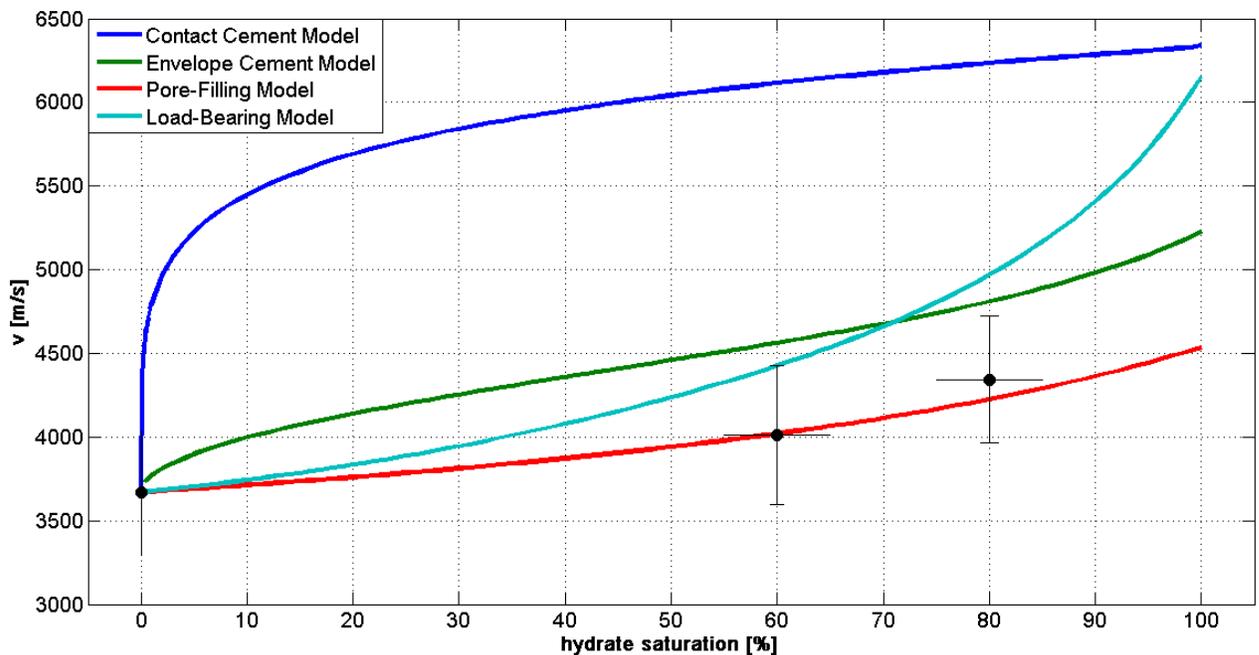


Figure 10: P-wave velocities for Bentheim sandstone samples with different hydrate saturations in comparison with effective medium model after Ecker et al. (1998) and Helgerud et al. (1999)

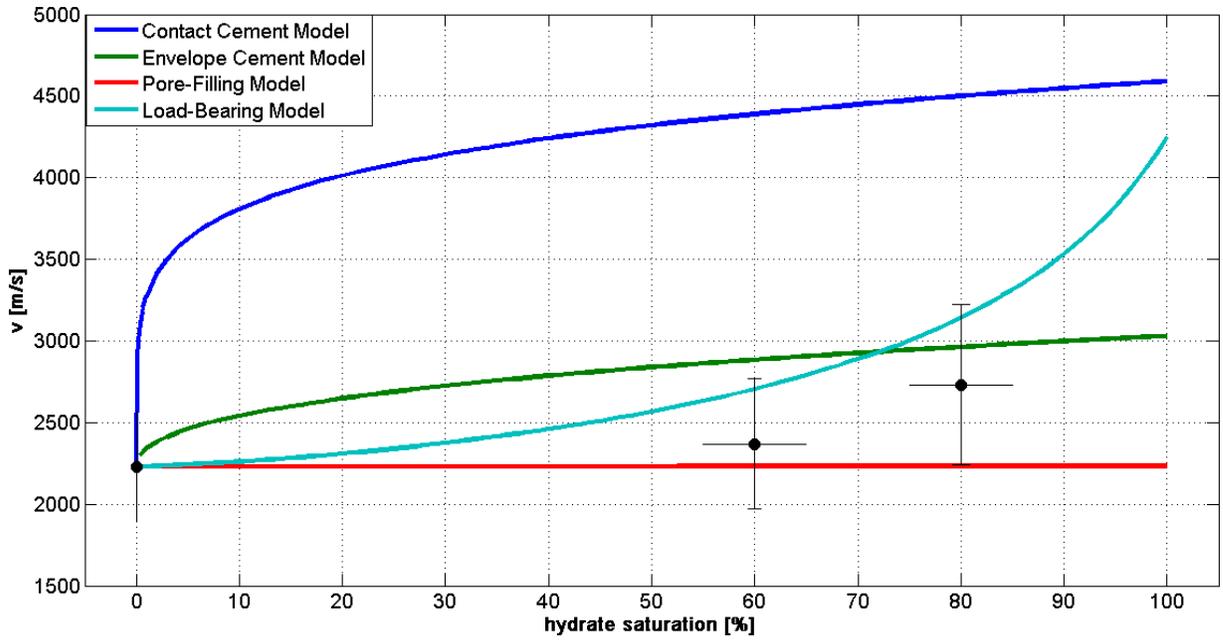


Figure 11: S-wave velocities for Bentheim sandstone samples with different hydrate saturations in comparison with effective medium model after Ecker et al. (1998) and Helgerud et al. (1999)

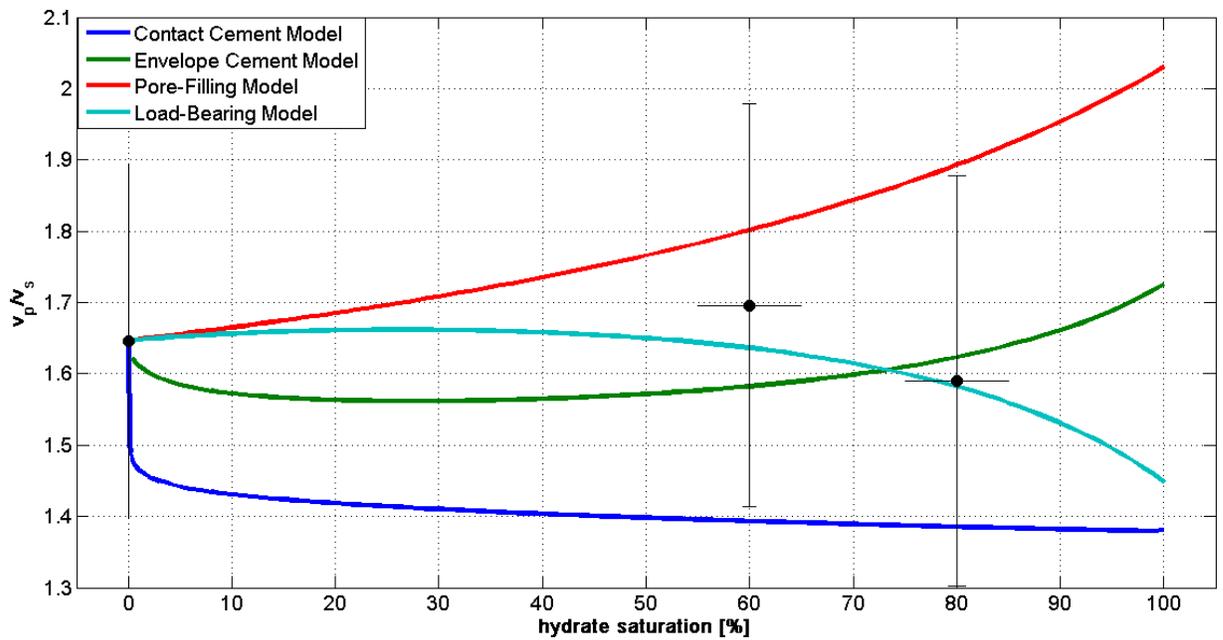


Figure 12: v_p/v_s ratio for Bentheim sandstone samples with different hydrate saturations in comparison with effective medium model after Ecker et al. (1998) and Helgerud et al. (1999)

The P-wave velocities coincide well with the pore-filling model while the S-wave velocities and the v_p/v_s ratio, respectively, are not conclusive yet. The data points lie between the envelope-cementing, pore-filling and load-bearing model. Additional measurements will be conducted for 20%, 40% and 100% hydrate saturation to show a clearer tendency towards one model.

Plans

We expect to create both methane and CO₂ hydrates in bulk in the near future. These bulk samples will allow us to develop the experimental conditions needed to impose. In addition, analytic procedures will be developed to characterize the hydrates themselves as well as evolved materials.

For attenuation measurements, the next steps involve the slope calculations of the divided spectra and compare those to the THF-water, THF-hydrate, “frozen” THF-hydrate, and pure ice. Also, NMR measurements have been done to determine if we have residual liquids in our sample after hydrate formation and if yes what kind of fluid it is. Our findings show that there might be some water molecules trapped in between the hydrate crystals. This needs to be more investigated and it also needs to be determined what the influence of this “residual” water is on our attenuation calculation for the 100 % THF-hydrate measurements at 1 °C. We have also collected shear waveforms and this need to be processed. We are assuming that these results will be more sensitive to residual water.

Further CT scans of THF-hydrate bearing Bentheim sandstone should give an insight into the distribution of hydrate in the pore space. The tomographies which were conducted so far were inconclusive due to very short scanning times. Short scanning times allow only very low resolution scans, longer scanning times resulted in blurring in the image due to hydrate dissociation. The average grain size of Bentheim sandstone is given as 200 μm (Stanchits et al., 2009) and is thus much smaller than the 1-mm-glass beads I was working on before. Therefore, a different way of cooling the sample has to be found in order to resolve hydrate in the pore space of fine grained, natural sediment.

DOE hydrates													
file name	date	scanning time [h]	host sediment	comments	magnification	source voltage	source power	exposure time	source position	detector position	number of images	angle increment	pixel size [μm]
JGR_Bentheim1_03-04-14x05	4-Mar-14	4.5	Bentheim Sst		0.5X	145 keV	9.5 W	0.5 s	-90 mm	26.8 mm	3601	+/-180°	0.1°
JGR_Bentheim1_03-04-14x20	4-Mar-14	45	Bentheim Sst		20X	145 keV	9.5 W	40 s	-42.664 mm	9 mm	3601	+/-180°	0.1°
Bentheim_dry_1	11-Mar-14	0.28	Bentheim Sst	determine parameters for THF scan	0.5X	150 keV	10 W	2 s	-54 mm	470 mm	191	+/-95°	1°
Bentheim_dry_2	11-Mar-14	0.87	Bentheim Sst	determine parameters for THF scan	0.5X	150 keV	10 W	2 s	-54 mm	470 mm	571	+/-95°	1/3°
Bentheim_dry_3	11-Mar-14	0.67	Bentheim Sst	determine parameters for THF scan	0.5X	150 keV	10 W	0.5 s	-54 mm	200 mm	601	+/-100°	1/3°
Bentheim_dry_4	11-Mar-14	1.42	Bentheim Sst	determine parameters for THF scan	4X	150 keV	10 W	10 s	-51 mm	20 mm	377	+/-94°	0.5°
Bentheim_dry_5	12-Mar-14	1.42	Bentheim Sst	determine parameters for THF scan	4X	150 keV	10 W	10 s	-51 mm	20 mm	377	+/-94°	0.5°
Bentheim_dry_6	12-Mar-14	1.42	Bentheim Sst	determine parameters for THF scan	4X	150 keV	10 W	10 s	-42.664 mm	10 mm	381	+/-95°	0.5°
Bentheim_water_1	13-Mar-14	1.42	Bentheim Sst	determine parameters for THF scan	4X	150 keV	10 W	10 s	-42.664 mm	13 mm	381	+/-95°	0.5°
Bentheim_water_2	13-Mar-14	9.25	Bentheim Sst	determine parameters for THF scan	4X	150 keV	10 W	15 s	-42.664 mm	13 mm	1801	+/-180°	0.2°
Bentheim_THF_hydrate1_tomo1	21-Mar-14	1.42	Bentheim Sst	hydrate dissociation, scan1	4X	150 keV	10 W	10 s	-51 mm	20 mm	377	+/-94°	0.5°
Bentheim_THF_hydrate1_tomo2	21-Mar-14	1.42	Bentheim Sst	hydrate dissociation, scan2	4X	150 keV	10 W	10 s	-51 mm	20 mm	377	+/-94°	0.5°
Bentheim_THF_hydrate1_tomo3	21-Mar-14	1.42	Bentheim Sst	hydrate dissociation, scan3	4X	150 keV	10 W	10 s	-51 mm	20 mm	377	+/-94°	0.5°
Bentheim_THF_hydrate2_tomo1	26-Mar-14	1.42	Bentheim Sst	hydrate dissociation, scan1	4X	150 keV	10 W	10 s	-51 mm	15 mm	377	+/-94°	0.5°
Bentheim_THF_hydrate2_tomo2	26-Mar-14	1.42	Bentheim Sst	hydrate dissociation, scan2	4X	150 keV	10 W	10 s	-51 mm	15 mm	377	+/-94°	0.5°
Bentheim_THF_hydrate2_tomo3	26-Mar-14	1.42	Bentheim Sst	hydrate dissociation, scan3	4X	150 keV	10 W	10 s	-51 mm	15 mm	377	+/-94°	0.5°
Bentheim_THF_hydrate3_tomo1	21-Mar-14	0.7	Bentheim Sst	hydrate dissociation, scan1	4X	150 keV	10 W	10 s	-51 mm	15 mm	377	+/-94°	0.5°
Bentheim_THF_hydrate3_tomo2	21-Mar-14	0.7	Bentheim Sst	hydrate dissociation, scan2	4X	150 keV	10 W	10 s	-51 mm	15 mm	377	+/-94°	0.5°
Bentheim_THF_hydrate3_tomo3	21-Mar-14	0.7	Bentheim Sst	hydrate dissociation, scan3	4X	150 keV	10 W	10 s	-51 mm	15 mm	377	+/-94°	0.5°
Bentheim_dry_fiberglass_vessel1	1-Apr-14	4.5	Bentheim Sst	test with CH4 tubing attached	0.5X	80 keV	4.2 W	1 s	-50 mm	100 mm	3601	+/-180°	0.1°
Bentheim_dry_fiberglass_vessel2	1-Apr-14	4.5	Bentheim Sst	test with CH4 tubing attached	0.5X	150 keV	10 W	1 s	-100 mm	300 mm	3601	+/-180°	0.1°
Bentheim_dry_fiberglass_vessel3	1-Apr-14	4.5	Bentheim Sst	test with CH4 tubing attached	0.5X	150 keV	10 W	1 s	-100 mm	300 mm	3601	+/-180°	0.1°
		90.37											

Table 1. Xray tomographic (CT) scans of the Bentheim sandstone under various saturation and mounting conditions

Participants and Collaborating Organizations

Name: George Radziszewski
Project Role: Research Faculty
Nearest person month worked this period: 1.5
Contribution to Project: Dr. Radziszewski spent his time establishing standards and procedures for running the MicroCT scanner .
Funding Support: "Organics, Clays, Sands and Shales (OCLASSH) consortium
Collaborated with individual in foreign country: No
Country(ies) of foreign collaborator: N/A
Travelled to foreign country: Yes
If traveled to foreign country(ies): Poland
Duration of stay: 2 weeks

Name: Mathias Pohl
Project Role: Graduate Student
Nearest person month worked this period: 3
Contribution to Project: Mr. Pohl prepared samples and collected ultrasonic data.
Additional Funding Support: N/A
Collaborated with individual in foreign country: No
Country(ies) of foreign collaborator: N/A
Travelled to foreign country: No
If traveled to foreign country(ies),
duration of stay: N/A

Name: Mandy Schindler
Project Role: Graduate Student
Nearest person month worked this period: 3
Contribution to Project: Ms Schindler prepared samples and collected CT data.
Additional Funding Support: N/A
Collaborated with individual in foreign country: No
Country(ies) of foreign collaborator: N/A
Travelled to foreign country: No
If traveled to foreign country(ies),
duration of stay: N/A

Name: Weiping Wang
Project Role: Laboratory Technician
Nearest person month worked this period: 0 (just started involvement)
Contribution to Project: Mr. Wang assisted in equipment fabrication
Additional Funding Support: N/A
Collaborated with individual in foreign country: No
Country(ies) of foreign collaborator: N/A
Travelled to foreign country: Yes

If traveled to foreign country(ies), N/A
duration of stay: N/A

Name: Michael Batzle
Project Role: Principle Investigator
Nearest person month worked: 1
Contribution to Project: Overall (dis)organization.
Funding Support: Academic faculty
Collaborated with individual in foreign country: No
Country(ies) of foreign collaborator: N/A
Travelled to foreign country: N/A
If traveled to foreign country(ies):

External Collaborations:
Dr. Tim Collett
US Geologic Survey
Denver, Colorado: (if foreign location list country)
Support: Data and guidance on interpretation and application
Tim continues to publish numerous papers on hydrate properties

Changes / Problems

Mr. Mathias Pohl will conduct an internship at shell Inc. for three months. However, no significant disruption of the project is anticipated.

Special Reporting Requirements

None

Budgetary Information

Attached separately

References

Ecker, C., J. Dvorkin and A. Nur (1998). "Sediments with gas hydrates: Internal structure from AVO." *Geophysics*, 93 (5): p. 1659-1669

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Society of Core Analysts held in Calgary, Canada

Stanchits, S., J. Fortin, Y. Gueguen, and G. Dresen (2009). "Initiation and Propagation of Compaction Bands in Dry and Wet Bentheim Sandstone." *Pure and Applied Geophysics*, 166, p. 843-868

Milestone Status

Our current position on the time chart is shown in Figure 13. We have approached the halfway mark of the project, and we are basically on schedule.

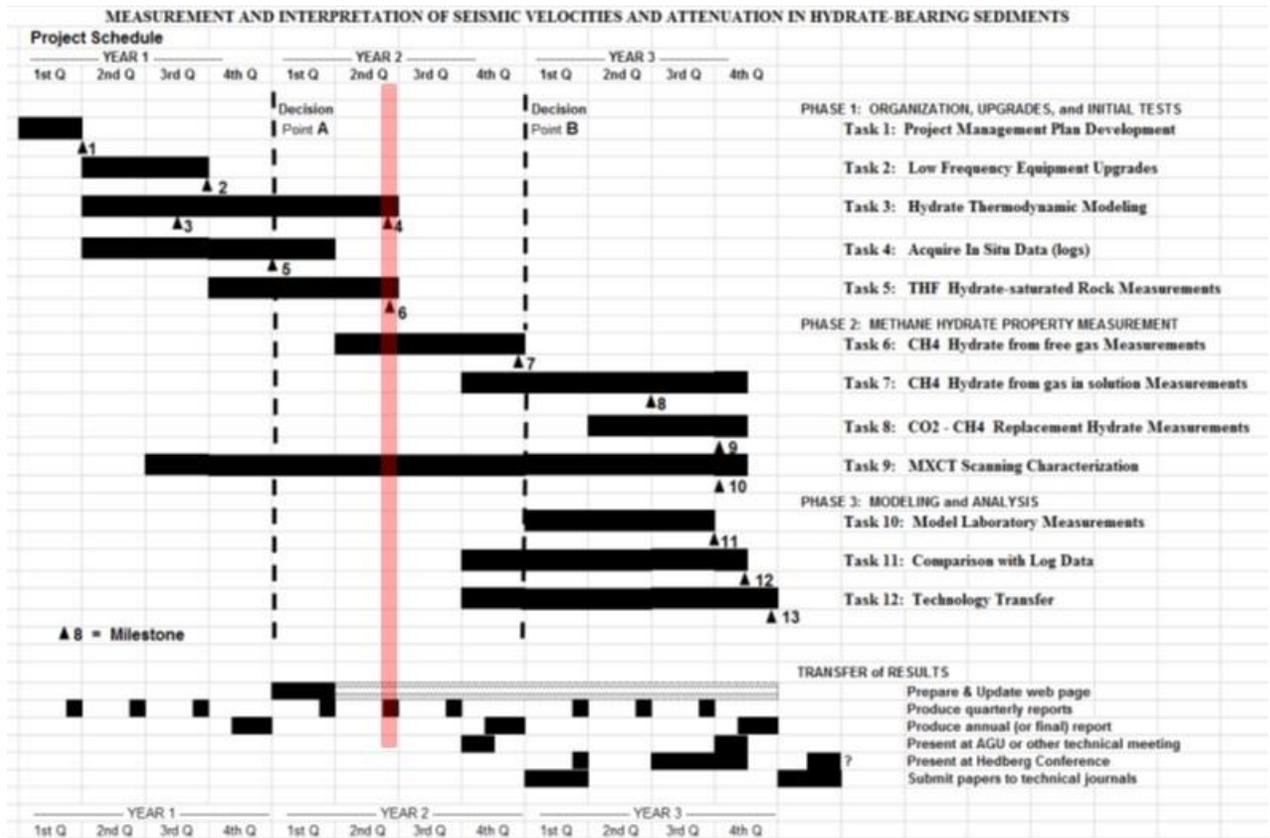


Figure 13. Milestone Status. We are at the end of our sixth quarter, near the midpoint of this project (red line)

In general, we are slightly behind on Milestone #5 (I need to fix the links in our website), and slightly ahead on Milestone #7 (we have grown some CH₄ hydrate).

Measurement and Interpretation of Seismic Velocities and Attenuations in Hydrate-Bearing Sediments
 DOE Award No.: DE-FE 0009963

		Planned Completion	Actual Completion	Verification	Comments
	Milestone Title / Description	Date	Date	Method	
1	Project Management Plan (PMP)	1-Dec-12	28-Nov-12	DOE acceptance	Complete and approved
2	Modifications to low frequency system	1-Jun-13	30-Jun-13	Data collected	Complete
3	Modeling established using EOS	31-May-13	30-Mar-14	Phase Diagr.	Complete
4	Property models of hydrates complete	31-May-13	30-Mar-14	Models plotted	Complete
5	Logs acquired and database estab.	31-Dec-13			Web needs update
6	THF hydrate grown in pressure vessel	1-Jun-14			Ahead of schedule
7	Methane hydrates from free gas phase	31-Dec-14			On Schedule
8	Methane hydrates from gas in solution	30-Jun-15			Planned
9	CO ₂ replacing methane in hydrates	30-Sep-15			Planned
10	MXCT scans completed	30-Sep-15			Continuing
11	Effective media models complete	30-Sep-15			Planned
12	Comparison to in situ data complete	15-Oct-15			Planned
13	Information Dissemination	31-Dec-15			Continuing