JAMSTEC Chikyu Expedition 906

"The Kumano Mud-Volcano Drilling II"

June 23 (Sasebo) to June 28 (Ugata), 2012



Preliminary Expedition Report

Fumio Inagaki, Yusuke Kubo and Expedition 906 Shipboard Scientists



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1. General Background

Submarine mud-volcanoes are globally distributed in plate convergent margins (Milkov 2000; Kopf 2002). The mud-volcanism supplies hydrocarbon gases and subsurface fluids from deep realm to the ocean surface, which therefore plays a significant role in the global fluid and element cycles (Etiope et al., 2009). The driving force and trigger of mud-volcano formation is generally associated with tectonic activity of plate subduction zone. Generally, the fluid from deep source contains hydrocarbons, metal elements and other gaseous components as well as brecciated sediments. On the seafloor of mud-volcano, cold seep-associated sediments harbor chemosynthetic life, including microbial communities that play significant ecological roles in biogeochemical cycle. For example, microbial activity that mediates anaerobic oxidation of methane coupled with sulfate reduction in the Haakon Mosby mud-volcano was found to consume approximately 40% of emitting methane (Niemann et al., 2006). Despite such unique geological and geochemical structures of mud-volcano as a "natural pipeline" that conveys very deep sedimentary matter to the surface ocean, the biogeochemical and microbiological nature of submarine mud-volcano subsurface has remained poorly understood.

The Nankai Trough off Kii Peninsula of Japan is one of the most geologically active plate subduction zones, where episodic gigantic earthquakes and tsunamis have historically occurred and resulted in serious disasters. Previous efforts on deep-sea research in the Nankai Trough showed that at least 10 mud-volcano-like conical-shape mounds are present in the Kumano forearc basin. Pilot geological and geochemical surveys have demonstrated that the Kumano mud-volcano no. 5 is one of the active mud-volcanoes in terms of the fluid and gas seepage, where the seep-associated chemosynthetic communities have been observed at near the summit.

During the Chikyu Training Expedition CK09-01 in 2009, we obtained

sediment core samples from the mud-volcano no. 5 to the depth of 20 meters below the seafloor (mbsf). Onboard observations using thermo-view IR camera and X-ray CT scan demonstrated that the cored sediment consisted mainly of dehydrated mud matrices and consolidated breccias, in which dendritically networked vertical formation of methane hydrates was also observed. Using a fluorescent image-based cell count technique, a relatively small population of bacterial and archaeal cells ($\sim 10^5$ cells/cm³) was observed, from which intact and amplifiable genomic DNA was extracted for the molecular ecological analyses. Interestingly, analyses of the pore water chemistry and its isotopic compositions showed the presence of extraordinary ¹³C-enriched bicarbonate in the sediment. This may be associated with metabolic activities of subseafloor microbial communities such as CO₂-reducing methanogenesis and/or acetogenesis. These preliminary microbiological and biogeochemical results suggest that the supply of nutrient and energy components through the mud-volcano subsurface harbors a unique habitat of subseafloor microbial community that may play important roles in biogeochemical carbon cycle.

One of the other significant scientific aspects relating to the submarine mud-volcanoes in the Nankai Trough is the relationship between the formation mechanism of deep-sourced fluids and gaseous components and the tectonics of accretionary wedge. Isotopic analysis of H_2O in sediment cored from the mud-volcano no. 5 indicated that the fluid water is generated from the smectite-illite dehydration, which process occurs in the temperature range between 60 °C and 160 °C. Based on the typical temperature gradient of the Kumano basin area, the depth range of possible dehydration process corresponds to the bottom of basin sediment or the upper accretionary prism, approximately at 2,500 mbsf. On the other hand, the subsurface sediment of the Kumano mud-volcano contains unusually high concentrations of hydrogen and carbon monoxide, which were found to be two or more orders of magnitude higher than those in the surrounding Kumano basin sediment. In addition, isotopic analysis of lithium (Li) indicates the potential source from the in-situ temperature of over 300 °C, close to the deep seismogenic fault zone.

Taken these geochemical and microbiological characteristics together with the geological setting of the Nankai Trough subduction zone, we can infer that the

mud-volcano in the Nankai Trough forearc basin is "the window to the deep seismogenic zone", in which geochemical and biological characteristics and behaviors may be highly sensitive to the seismogenic fault activity. Hence, the long-term monitoring of such subsurface geochemical and geophysical parameters of the submarine mud-volcano subsurface through the borehole may be a good strategy to find the way towards the better understanding of earthquake and tsunami-related geological movements, including the stress and friction that may cause geochemical and geophysical anomalies.

To expand our knowledge of the submarine mud-volcano subsurface, we planned to drill the previous site on the Kumano mud-volcano no. 5 down to 200 mbsf using a combined use of a hydraulic piston coring system (HPCS) and a newly developed hybrid-pressure coring system (Hybrid-PCS) on *Chikyu* during the JAMSTEC Expedition 906. The shipboard scientific party of Expedition 906 is organized by multidisciplinary expertise including geophysics, sedimentology, physical property, geochemistry, microbiology and molecular microbiology. Despite the relatively short duration of the cruise, some of the planned operations and analyses are the first attempt and hence challenge, opening a new window to the deep subseafloor biosphere and seismogenic zone.

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2. Scientific Objectives

The main research objectives during Expedition 906 "The Kumano mud-volcano drilling II" are as follows:

- 1. To characterize in-situ geochemical and geophysical characteristics of solid, gas and fluid components in deep mud-volcano subsurface,
- To explore the deep subseafloor biosphere within the mud-volcano and evaluate the population, diversity and metabolic activity of microbial community and its physiological state and energetic habitability,
- 3. To evaluate the impact of subseafloor microbial activities in biogeochemical cycles of the plate accretionary wedge, and
- 4. To understand the nature and amount of methane hydrates and characterize the formation mechanism of the hydrocarbon reservoir in the mud-volcano diapir.

To achieve these major scientific objectives, we drill at Site C9004 Hole A, the same location that was previously drilled down to ~20 mbsf in 2009 during the *Chikyu* training cruise CK09-01, and we extend the coring depth down to ~200 mbsf. To explore the deep mud-volcano subsurface, keeping in-situ pressure is critical for the analysis of methane hydrates and volatile gas components such as H₂ and CO, which have not been accurately evaluated by the standard hydraulic piston coring system (HPCS) despite the tremendous scientific significance. To address the issue for the deep mud-volcano subsurface environment, we newly developed a hybrid pressure coring system (Hybrid-PCS) that is available for the *Chikyu* and also compatible with the GeoTek Pressure Core Analysis and Transfer System (PCATS). In addition, to estimate microbial activity of the deep subseafloor biosphere, we deployed a new container lab (ISOVAN) for the radioactive tracer experiments.

3. Location of Drilling Site

The drilling site location during Expedition 906 is located on the southeastern slope near the summit of Kumano mud-volcano no. 5. We drilled ~200 meters at Site C9004 Hole B, moved 10 meters west from Hole B, and drilled ~6.5 meters at Site C9004 Hole C.(See, **Table 3.1**, **Figs. 3.1**, **3.2** and **3.3**).

Table 3.1. Ex	pedition	906 Lc	ocation	Summary.
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Site	Hole	Coo	inates		I	Depth
	11010	La itude	Longitude		(mBRT)	(mbsf)
	В	33°40.5486'N	136°34.0851'E	1900.0	2132.0	203.5
	С	33°40.5486'N	136°34.0786'E	1905.0	1940.0	•

leva io



Fig. 3.1. Location map of the drilling site, the Kumano mud-volcano no. 5. off Kii Peninsula of Japan.



Fig. 3.2. Mud-volcanoes in the Kumano forearc basin of the Nankai Trough.



Fig. 3.3. Location of Site C9004 Hole A, B, and C on the mud-volcano no. 5.

4. Drilling Operation

Please input data at Red character



Fig. 4.1. Water depth and mud-line core summary for Site C9004 Hole B.

Table 4.1. Expedition 906 Core Recovery Summary

		Onvine	Total	Total Cored	Total Initial	Total Curated	Total Corrected	Initial	Corrected	Water Depth	
Site	Hole	Coring	Advance	interval	Core Length ¹⁾	Liner Length	Core Length ²⁾	Recovery ³⁾	Recovery4)	(mBSL)	(mBRT)
		System	(m)	(m)	(m)	(m)	(m) (m)		(%)	(IIIDOL)	(IIIDITT)
		HPCS	-	34.8	42.39	46.72	40.04	121.8	115.0		
		ESCS	-	9.5	1.27	1.27	1.27	13.4	13.4		
C0004	В	HYB-EP	-	7.0	0.25	0.40	0.23	3.6	3.3	1900.0	1928.5
09004		HYB-ES	-	8.0	0.20	0.00	0.00	2.5	0.0		
		Total	203.5 59.3		44.11	48.39	41.54	74.4	70.0		
	С	HYB-ES	6.5	6.5	0.90	0.90	0.90	13.8	13.8	1905.0	1933.5

Measured by curatorial staff at Core Cutting Area (not same with the final curated length on J-CORES)
 Curated liner length - Void space and extraneous material (flow-in, cavings, fills, cuttings etc)
 Curated liner length / advance (final record on J-CORES)
 Curated length (rest)

4) Corrected core length / advance





Fig. 4.2. Core recovery of Site C9004 Hole B.

Coring Depth Management Sheet for Expedition 906 (CK12-03 Leg 1) Kumano #5 Knoll Drilling II

Water Depth :	1900.00	mMSL	= 1928.50 mBRT
Elevation :	28.50	m	Elevation=Length(Rotary Table ~ Bottom of ship)-Draft
Coring System Code	•		
H: HF	CS: Hydraulic	Piston Coring	g System
T: EP	CS: Extended	Punch Coring	ig System
X: E5	CS: Extended-	Shoe Coring	g System
D. HY	B. Hybrid Dree	euro Coro Se	ampler



Position Proposed										
Lat:	33°40.5486'N									
Lon:	136°34.0851'E									
Position Actual										
Position /	Actual									
Position / Lat:	Actual 33°40.5486'N									

1) Determined based on hit denth with taking HPS torque and WOR into account at the time in case of RCR_ESCS and EPCS

¹² Contracted Carle Lendth / Advance (final record on .I-CORES)
¹⁰ Carreted Care Lendth / Advance (final record on .I-CORES)
¹⁰ Lendth of null-up of drill string until taking over weight in case of partial penetration of HPCS

				Coring		Below Ro (mi	otary Table BRT)	Below Seafloor (mbsf)			Initial		Initial	Coring	Average	Extraneo	us Material (m)	Intervals ⁵⁾	Curated	Corrected	Corrected	Overpull	
Core	Tuno	adf.	(JST) 1)	Termination Time (JST)	Core on Deck Time (JST)	Top of Core	Btm of core ⁹⁾	Top of Core	Btm of Core ⁹⁾	Advance ²⁾ (m)	Core Length ³⁾ (m)	Lithology @ shoe	Recovery4) (%)	Time (min)	ROP (m/hour)	Upper Portion (fills, cavings)	Lower Portion (flow-in, cuttings)	Total Void Portion	Core Length ⁸⁾ (m)	Core Length ^{s)} (m)	Recovery ⁷⁾ (%)	Length ⁹⁾ (m)	Remarks
1	н		2012.06.25 21:15	2012.06.25 21:15	2012.06.25 22:52	1923.00	1932.50	-5.50	4.00	9.50	9.68	Greenish mid-gray clay bearing granules	101.9	0				5.80	9.80	4.00	42.1		w/ APCT-3. Blew off from the top with carbonate/concretion pebbles and mud. Highly expanded.Greenish mid-gray pebbly clay. H2S detected. Mudline was determined after XCT scan.
			2012.06.26 00:30	2012.06.26 00:57		1932.50	1943.00	4.00	14.50	10.50				27	23.3								Drill down
2	н		2012.06.26 02:07	2012.06.26 02:07	2012.06.26 02:58	1943.00	1950.70	14.50	22.20	7.70	7.72	Greenish mid-gray clay bearing granules	100.3	0				0.25	7.80	7.56	98.1	-	w/ APCT-3. Partial penetration, picked up w/ coreline. Blew off from the top with carbonate/conretion pebbles and mud. Greenish mid-gray Pebbly clay.
			2012.06.26 03:28	2012.06.26 03:30		1950.70	1952.50	22.20	24.00	1.80				2	54.0								Drill down
3	Ρ	л	2012.06.26 07:39	2012.06.26 07:41	2012.06.26 08:33	1952.50	1955.50	24.00	27.00	3.00	0.05	Mid-gray soupy mud w/ granules	1.7	2	90.0			0.10	0.15	0.05	1.7		Hybrid-PCS w/ EPCS-type shoe. Failed to keep in- situ pressure due to unproper function of check valve.
			2012.06.26 08:55	2012.06.26 09:26		1955.50	1978.50	27.00	50.00	23.00				31	44.5								Drill down w/ kelly down once
4	н		2012.06.26 10:59	2012.06.26 10:59	2012.06.26 11:45	1978.50	1987.50	50.00	59.00	9.00	9.75	light olive gray sandy clay w/ granules	108.3	0				0.34	9.74	9.40	104.4	0.5	w/ APCT-3. Partial penetration. Blew off from top and bottom. Highly expanded. Light olive gray to greenish mid-gray Pebbly clay.
			2012.06.26 12:38	2012.06.26 12:39		1987.50	1988.00	59.00	59.50	0.50				1	30.0								Drill down
5	Ρ	л	2012.06.26 22:15	2012.06.26 22:29	2012.06.26 23:00	1988.00	1992.00	59.50	63.50	4.00	0.20	Greenish mid-gray sandy clay w/ pebbles	5.0	14	17.1			0.17	0.40	0.23	5.8		Hybrid-PCS w/ EPCS-type shoe. Sample only at CC. Succeeded to keep almost in-situ pressure.
			2012.06.27 00:11	2012.06.27 00:53		1992.00	2028.50	63.50	100.00	36.50				42	52.1								Drill down w/ ESCS, kelly down once
6	н		2012.06.27 03:22	2012.06.27 03:22	2012.06.27 03:59	2028.50	2034.00	100.00	105.50	5.50	6.10	Greenish mid-gray sandy clay w/ granules	110.9	0				0.10	5.88	5.78	105.1	4.3	w/ APCT-3 . Partial penetration. Extruded from the top about 2.4 m long. Blew out from the bottom. Highly expanded. A piece of hydrate at near bottom. APCT-3 measurement failed due to water leak.
			2012.06.27 04:16	2012.06.27 04:20		2034.00	2038.00	105.50	109.50	4.00				4	60.0								Drill down w/ ESCS
7	Ρ	ſΧ	2012.06.27 11:41	2012.06.27 11:46	2012.06.27 12:34	2038.00	2042.00	109.50	113.50	4.00	0.20	Greenish mid-gray sandy clay w/ granules	5.0	5	48.0				0.20	0.20	5.0		Hybrid-PCS w/ ESCS-type shee. Roughly 0.05 m recovery in CC. Based on PCAT data, about 0.2 m core is recovered though in-situ pressure is not preserved. This recovery data is based on first observation for X-CT data from PCAT. Actual samaple might be slurred mud.
8	н		2012.06.27 14:14	2012.06.27 14:14	2012.06.27 15:07	2042.00	2050.60	113.50	122.10	8.60	9.14	Greenish mid-gray sandy clay w/ granules	106.3	0				0.20	13.50	13.30	154.7	0.9	w/ APCT-3. Retrial for APCT-3. Pebbly mud.
			2012.06.07 16:45	2012.06.27 18:00		2050.60	2118.50	122.10	190.00	67.90				28875	0.1								Drill down w/ ESCS
9	Ρ	١X	2012.06.27 22:23	2012.06.27 22:29	2012.06.28 23:41	2118.50	2122.50	190.00	194.00	4.00	0.00	empty	0.0	6	40.0				0.00	0.00	0.0		Hybrid-PCS w/ ESCS-type shoe. Suceeded to keep in-situ pressure, but no recovery.
10	x		2012.06.28 01:15	2012.06.28 01:20	2012.06.28 03:04	2122.50	2132.00	194.00	203.50	9.50	1.27	Dark greenish gray mudstone breccia	13.4	5	114.0				1.27	1.27	13.4		CC plugged. Alternation of mudstone breccia and pebbly mud.
			N Total Co	umber of Coring: red Advance (m):	10 64.80		Total Initi Average I	al Core Le nitial Reco	ngth (m): very (%):	44.11 68.07		Total Corrected Core Average Corrected Re	Length (m):	41.79 64.5									

Total Corrected Core Length (m): 41.79 Average Corrected Recovery (%): 64.5

Coring Depth Management Sheet for Expedition 906 (CK12-03 Leg 1) Kumano #5 Knoll Drilling II







Lon:

136°34.0786'E

¹⁾ Determined based on bit denth with taking HPS torque and WOB into account at the time in case of RCB_FSCS and FPCS

²⁾ For HPCS coring with incomplete shooting, determined by subtracting the remained length of telescope extension from 9.5 m or core recovery in case of no indication of over weight while pulling up the telescope. Shall be finally determined after checking if there is flow-in structure by the XCT image.

³⁾Measured by curatorial staff at Core Cutting Area (not same as the final curated length on J-CORES)

4) Initial Core Length / Advance

⁵⁾ Confirm with XCT image and/or solit core. and calculate the length from XCT image on Composite Log Viewer of J-CORES

6) Curated Core Length - Exraneous Material (flow-in. cavings. fills. cuttings. void space) Interval Length

7) Corrected Core Length / Advance

⁸⁾Curated Core Length / Advance (final record on J-CORES) 9) Length of pull-up of drill string until taking over we eight in case of partial penetration of HPCS

		Caring Start Time	Coring		Below Rotary Table (mBRT)		Below Seafloor (mbsf)		Initial	Ir	Initial	Initial Coring	Average	Extraneous Material Intervals ⁵⁾ (m)		Curated	Corrected	Corrected	Overpull			
Core	Type	(JST) 1)	Termination Time (JST)	Core on Deck Time (JST)	Top of Core	Btm of core ⁹⁾	Top of Core	Btm of Core ⁹⁾	Advance ²⁾ (m)	Length ³⁾ (m)	Lithology @ shoe	Recovery ⁴⁾ (%)	Time (min)	ROP (m/hour)	Upper Portion (fills, cavings)	Lower Portion (flow-in, cuttings)	Total Void Portion	Length ⁸⁾ (m)	Length ⁶⁾ (m)	Recovery ⁷⁾ (%)	Length ⁹⁾ (m)	Remarks
1	P /X	2012.06.28 08:47	2012.06.28 08:56	2012.06.28 09:27	1933.50	1940.00	0.00	6.50	6.50	0.90		13.8	g	43.3				0.90	0.90	13.8		Hybrid-PCS w/ ESCS-type shoe. Succeeded to keep the set pressure (3265psi). Recovered an intact cylindrical core. Rich in vein-like feature (hydrate?).
		N.	umbor of Coring:	4		Tatal Initi	al Canal a	math (ma).	0.00		Total Courseted Cours	I am math (ma).	0.00									

Number of Coring: 1 Total Cored Advance (m): 6.50

Total Initial Core Length (m): 0.90 Average Initial Recovery (%): 13.85

Total Corrected Core Length (m): 0.90 Average Corrected Recovery (%): 13.8

5. Core Flow

5.1. Hydraulic Piston Coring System (HPCS) Sampling

Kumano Mud Volcano HPCS core Onboard Measurement Plan



Spectrophotometer Ion Chromatograph

5.2. Hybrid-Pressure Coring System (Hybrid-PCS)

Kumano Mud Volcano Hybrid PCS core Onboard Measurement Plan



6. Preliminary Onboard Results

6.1. Sediment temperature, thermal conductivity and gas hydrate stability

reported by Tomas Feseker and Yojiro Ikegawa

6.1.1. Background

The physical parameters that limit the occurrence of gas hydrates in the sub-seafloor are pressure and temperature. The drill site at the Kumano mud-volcano no. 5 is located at around 1900 m water depth, hence the pressure at the seabed is around 19.227 MPa at standard atmospheric pressure (101 325 Pa). According to an equation proposed by Tishchenko et al. (2005), pure methane hydrates are stable at this depth at temperatures below 17.46 °C, assuming a salinity of 20 PSU. The thickness of the gas hydrate stability zone (GHSZ) below the seabed depends on the sediment temperature profile. The temperature at the seabed is estimated at around 2 °C. Based on long-term temperature observations at shallow sediment depths, Hamamoto et al. (2011) reported sediment temperature gradients of 0.0444 ± 0.0011 °C/m and 0.0876 ± 0.0009 °C/m at around 2080 m water depth approximately 7.4 km east and 8.7 km south of the drill site, respectively. If the sediment temperature gradient were in the same range, the base of the GHSZ would be expected at depths of between 162 and 482 m below the sea floor (Fig. 6.1.1). A seismic line across the mud-volcano no. 5 shows a bottom simulating reflector (BSR) around 411 to 463 meters below the seafloor (mbsf) in the immediate vicinity, which suggests a regional background temperature gradient of between 0.0383 and 0.0426 °C/m (Fig. 6.1.2). Depending on the activity of the mud volcano, however, it may be expected that the upward advection of fluids and mud lead to a local increase in temperature gradient around the mud volcano, which would reduce the thickness of the GHSZ: e.g., if the strong seismic reflector at between 191 and 215 mbsf within the mud volcano marked the base of the GHSZ (Fig. 6.1.2), the sediment temperature gradient would have to be between 0.0766 and 0.0857 °C/m or approximately twice the background value. Consequently, precise in situ temperature measurements are of crucial importance in determining the section of the sediment column, in which gas hydrates may occur.

The heat flux through the sediment is defined as the product of temperature

gradient and thermal conductivity. The latter depends on the composition of the bulk sediment, and in particular on its porosity. In case the heat flux is constant and there is no advection of fluids, the thermal conductivity of a given interval controls the temperature gradient. On the other hand, advection of fluids within the pore space or mass movement of the bulk sediment may also affect the temperature gradient and result in curved temperature profiles. In contrast to any movements, the thermal conductivity of the sediment can be measured in the laboratory, which provides useful constraints for the interpretation of non-linear sediment temperature profiles.

6.1.2 Methods

During Expedition 906, *in situ* temperature measurements were conducted using the APCT-3 tool (Heesemann et al., 2006). Attached to the tip of the Hydraulic Piston Coring System (HPCS), the APCT-3 will record temperature readings at a sampling interval of 1 s during the each deployment of the HPCS. Measurements were obtained by stopping the drill bit above the seabed (mudline) and at selected depths for around 10 minutes. The equilibrium sediment temperatures were calculated by extrapolation from the time series recorded during the measurements (Villinger and Davis, 1987). The depths of the measurements were estimated from the depth of the drill bit and the core recovery. The thermal conductivity of the sediment was measured using needle probes on the whole round cores after equilibration with room temperature.

The dissociation temperature of pure methane hydrates was calculated according to Tishchenko et al. (2005), assuming a salinity of 20 PSU and standard atmospheric pressure (101325 Pa). The conversion between depth and pressure followed Saunders (1981).

6.1.3. Preliminary results

(1) Temperature at the seabed

In the course of the first deployment of the HPCS (C9004-B-1), the corer was stopped a few meters above the seabed in order to measure the bottom water temperature above the drill site prior to penetration (**Fig. 6.1.3**). After the pumps had been stopped, the temperature readings dropped by around 0.1 °C. A

corresponding temperature increase at the end of the measurement indicated that the pumps had been switched back on. The bottom water temperature of approximately 1.88 °C was determined as the mean value of the readings in between these to events. The cause of the relatively large fluctuations of the temperature readings around the mean is unclear.

(2) In situ sediment temperature and gas hydrate stability

Using the APCT-3 tool, *in situ* sediment temperature measurements were conducted at approximately 4, 22, 59, and 121 mbsf. The quality of the raw data was generally good. For each measurement, a time window of several minutes could be extracted from the recorded time series for extrapolation of the equilibrium temperatures (**Fig. 6.1.4**). The corresponding sediment temperature profile (**Fig. 6.1.5**) is curved convex upward in the upper part and becomes linear towards the bottom with a gradient of around 0.029 °C/m. Pure methane hydrates are stable throughout the profile. Assuming that the profile remains linear at greater depths, the estimated depth of the lower limit of the GHSZ is expected at around 590 mbsf.

(3) Thermal conductivity

The quality of thermal conductivity measurements depends on the availability of intact sediment samples. Cracks or gas bubbles in the sediment and large voids within sections of the cores prevent reliable measurements. Therefore, measurements were conducted only on apparently intact intervals of the sediment cores. The data suggest a trend from just below 1 W/(°C m) near the seabed to around 1.2 W/(°C m) at 120 mbsf (**Fig. 6.1.6**).

6.1.4 Summary and onboard conclusions

In situ sediment temperature measurements and thermal conductivity measurements of intact core sections where conducted in order to determine the thermal regime of the Kumano mud-volcano no. 5 and to determine the extent of the GHSZ. The sediment temperature profile is curved convex upward above around 60 mbsf and linear with a gradient of around 0.029 °C/m. The measured values of sediment thermal conductivity suggest a slight increase with depth, but

the trend is insufficient to explain the curvature in the upper part of the temperature profile. Assuming that the profile reflects steady-state conditions, the curvature could indicate upward advection of pore fluids in the order of 0.1 to 0.3 m/year. It is unclear however, why the temperature gradient at depth appears to be lower than in the surrounding seafloor.

The pressure and temperature conditions permit the formation of gas hydrates throughout the entire length of the profile and down to a depth of around 590 mbsf. Considering that the mud volcano is elevated around 100 m above the surrounding seabed, this depth matches the estimated depth of the regional BSR. The strong seismic reflectors observed within the mud volcano cannot be related to gas hydrate stability. Visual inspection of CT images of the sediment core recovered from around 190 m (C9004B-10X) suggests that the impedance contrast may be related to a layer containing large and massive clasts.

6.1.5. References

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- Villinger, H., and Davis, E. E. (1987), A New Reduction Algorithm for Marine Heat Flow Measurements, *J. Geophys. Res.*, **92**, 12 846-12 856.



Fig. 6.1.1. Estimated thickness of the gas hydrate stability zone (GHSZ) for pure methane hydrates at the drill site on Kumano mud-volcano no. 5 (1900 m water depth, 2 °C bottom water temperature, salinity 20 PSU, sulfate-free porewater).



Fig. 6.1.2. A SE-NW seismic line across the Kumano mud-volcano no. 5. shows a bottom simulating reflector (BSR) at around 411 to 463 m below the sea floor in the immediate vicinity of the mud volcano. Within the mud volcano, another strong reflector is located at around 191 to 215 m below the sea floor. Time to depth conversion for is based on p-wave-velocities of 1500 m/s in the water column, and 1600 to 1800 m/s in the sediment. (Seismic data courtesy of J. Ashi, University of Tokyo, Japan)



Fig. 6.1.3. Temperature measurement in the bottom water of the drill site using the APCT-3 tool. The bottom water temperature of around 1.88 °C was determined as the mean of the values recorded after the pumps of the corer had been switched off and the temperature had dropped by around 0.1 °C and before the temperature increase the indicated that the pumps had been switched on again. After the measurement, the Hydraulic Piston Coring System (HPCS) was shot into the sediment to obtain the mudline core.



Fig. 6.1.4. In situ sediment temperature measurements using the APCT-3 tool at approximately 4, 22, 59, and 121 mbsf. The dashed lines mark the limits of the time windows that were used to extrapolate the equilibrium temperatures, which are indicated by the horizontal red lines.



Fig. 6.1.5. The sediment temperature profile at the Kumano mud-volcano no. 5 is curved convex upward in the upper part and becomes linear at depth, indicating a temperature gradient of around 0.029 °C per m.



Fig. **6.1.6**. Thermal conductivity of the sediments at the Kumano mud-volcano no. 5, measured using a needle probe in the laboratory on board. The values suggest a trend from just below 1 W/(°C m) at the seabed to around 1.2 W/(°C m) at 120 mbsf.

6.2. Sedimentology

reported by Koichi lijima and Yuki Nakamura

6.2.1 Visual Core Description

Visual Core Description (VCD) was carried out using archive half section after image scanning (MSCL-I) of the core was finished. Both in MSCL-I and VCD, the surface of the cores was not scraped due to difficulty to clear the small undulation arise from heavy disturbance or scattering/intercalating firm mudstone clasts.

In the VCD, lithology (e.g. grain size, distribution, thickness, and characteristics), sedimentary structure, color (using munsell color chart), and drilling disturbances or cracks were recorded on description sheet. After that, all VCD information was input to J-CORES database through "*J-CORES Visual Core Description*" application.

6.2.2. Summary of lithology of Hole C9004B

Major lithology of Site C9004 Hole B is clay or silty clay, containing various size (granular to cobble) of mudstone clasts. Summary of each cores are as follows:

- Core 1H (Fig. 6.2.1): Homogenous gray clay and silty clay. The core is heavily disturbed and 1-3 mm size bubbles are scattered throughout the core. Sections 1-4 were almost empty but the wall inside the inner tube is totally covered by muddy sediments. These sediments might be derived from lower section due to expansion during HPCS recovery.
- **Core 2H** (**Fig. 6.2.2**): Homogenous gray silty clay containing mudstone clasts. The core is disturbed and 1-3 mm size bubbles are scattered throughout the core. There are some voids caused by gas expansion.
- **Core 4H** (**Fig. 6.2.3**): Homogenous gray clay to silty clay. The core is disturbed and 1-3 mm size bubbles are scattered throughout the core.
- **Core 6H** (**Fig. 6.2.4**): Homogenous gray silty clay. Sections 1-3 of this core was popped off on drill floor due to gas expansion and the parts were retrieved into core liner. So the voids and cracks in these sections were

artificially made.

- **Core 8H (Fig. 6.2.5):** Homogeneous gray clay, but pebble size mudstone clast is abundant rather than shallower cores. The core is heavily disturbed but mm size bubbles are not recognized.
- Core 10X (Fig. 6.2.6): Homogenous gray mudstone or brecciated mudstone. The core is heavily disturbed. Upward grading observed in section 1 might be developed while Extended Shoe Coring System (ESCS) drilling.

6.2.3 Smear-slide method

Some smear-slides were made (at least one per core) and observed using polarized microscope to recognize components and texture of the sediments for determination of the lithological name. Typical views of the smear-slide in Core 1H are shown in **Figs. 6.2.7** and **6.2.8**.

906-C9004B-1H; -5.5-4.1

3.000-

3.500

4.000-

8

cc No. _ _ _ _ _ _

and and

1H-7, 50.0 cm.

1H-CC, 0.0 cm.

1H-CC, 24.5 cm.

Silty clay. visual description. 1H-7, 100.0 cm.

Silty clay. visual description.



副

Fig. 6.2.1. Summary of VCD for Core 1H



depth section X ray CT scan (s section photo lithology distribution Depth DSF, I Se... Se... Lithologic a... Drilling Distu... 200 400 600 8 0.5 1.0 1.5 2.0 0.0 2 က နက်လ်မယ်နှ 14.500 2H-1, 0.0 cm. 00 FB. >€ B Clay, visual description. Unconsolidated (soft). Sediment components: Pelagic grain, 6%; Calcareous skeletons, 6%; Nannofossils, 6%; 15.000r enging grain, 9%, canceredus sketerons, 5%; Nannotossils, 5%; Siliciclastic grain, 88%; Minerals, 66%; Quartz, 6%; Clay mineral, 60%; Lithic grain, 22%; Sedimentary lithic grain, 22%; Others, 6%; Gypsiferous grain, 4%; Opaque grain, 2%. Sediment texture: Silt, 19%; Mud, 100%; Clay, 61%. 15.500-2H-1, 51.0 cm. 16.000-_____ 2H-2, 130.0 cm. 16.500-Silty clay. visual description. 2H-2, 139.4 cm. 17.000-17.500-2H-4, 0.0 cm. and the second s 18.000-Silty clay. visual description. 18.500-2H-4, 91.0 cm. ____ 19.000-2H-5, 30.0 cm. Le Romanne and Andream 19.500 Silty clay. visual description. 4.4. Band S 20.000-2H-6, 0.0 cm. -6 2H-5, 141.0 cm. ____ 20.500-Clay. visual description. 出 2H-6, 17.0 cm. 2H-6, 27.0 cm. 21.000-Clay. visual description. 2H-6, 60.0 cm. 2H-7, 0.0 cm. 21.500-Clay, visual description. , B 2H-7, 42.0 cm. 22.000-2H-CC, 0.0 cm. ----_____ Silty clay. visual description.

sedimentary structure distribu drilling disturban GRA density (g/cm³) Magnetic susceptibility : P-wave velocity (m/s)

8

2H-CC, 35.5 cm.

Fig. 6.2.2. Summary of VCD for Core 2H.

906-C9004B-4H; 50.0-59.37



sedimentary structure distribu drilling disturban GRA density (g/cm³) Magnetic susceptibility : P-wave velocity (m/s)

Fig. 6.2.3. Summary of VCD for Core 4H.

906-C9004B-6H; 100.0-105.895

depth section X ray CT scan (s section photo lithology⊡distribution Depth DSF, I





Fig. 6.2.4 Summary of VCD for Core 6H.

906-C9004B-8H; 113.5-122.73

depth section X ray CT scan (s section photo lithology::distribution Depth DSF, I

sedimentary structure⊟distribi drilling⊡disturban GRA density (g/cm³) Magnetic susceptibility : P-wave velocity (m/s) S... S... Lithologic acce... Drilling Distu...


Fig. 6.2.5. Summary of VCD for Core 8H





37

Fig. 6.2.6 Summary of VCD for Core 10X.



x200 cross nicol

Fig. 6.2.7. Smear-slide photo image of hole C9004B, Core 1H-1, interval 75cm. Nannofossils and small (less than 1 micrometer) carbonate grains are common through the hole.



x200 cross nicol

Fig. 6.2.8. Smear-slide photo of hole C9004B, Core 1H-1, interval 75cm. Authigenic dolomite grains are contained.

6.3. Syringe and Whole Round Core (WRC) Sampling

reported by Tatsuhiko Hoshino, Rhishi Ram Adhikari, David Case, Yuki Morono, Hans Røy, Sakai Sanae, Takeshi Terada, Hitoshi Tomaru, Takayuki Tomiyama, and Nan Xiao

6.3.1. Sampling Procedures

(1) Syringe sampling

All syringe sampling was done in the core cutting area immediately after core arrival. Generally the syringe samples were taken from section tops at a frequency of 1 sample/section. The exception was the JKHS code sample, which was taken from section bottoms at a frequency of 1 sample/core. The sampling flow is shown in **Fig. 6.3.1.** Specific sampling positions and other details are described in **Appendix 1**, including deviations from the top/bottom sampling plan described above.

- ATHS: 3ml of the sediment was extracted by 2.5 ml syringes, put in plastic bag and stored at 4 °C.
- **DCHS1:** 3ml of the sediment was extracted by 3 ml syringes, ejected into vials with 2 mL NaOH, crimped and stored at room temperature (RT).
- **DCHS1:** 5ml of the sediment was extracted by 5 ml syringes, ejected into vials with 5 mL NaOH, crimped and stored at RT.
- AIHS1: 3 ml of the sediment was extracted by 2.5 ml syringes. Only the top of 2.5 ml sediment was ejected into glass bottle with NaCl to remove contamination from the surface of sediment. The samples were stored at RT.
- AIHS2: 3 ml of the sediment was extracted by 2.5 ml syringes. After 0.5 ml of sediment was removed, the syringes were plugged with rubber plug to avoid exposure of oxygen. The samples were stored at RT.
- YMHS: ~10 ml of the sediment was extracted by using 10 ml syringe. Only first 5 ml of the sediment was ejected to avoid contamination from the surface of sediment into 25 ml of 4% PFA in 50 ml Falcon tubes. In the microbiology lab, the samples were mixed until the sediment was completely suspended. The suspension was stored at 4 °C at least 6

hours for fixation. After the fixation, the sediment was centrifuged at $5,000 \times g$ for 15 min and the supernatant was removed. The fixed sediment was washed with 1 x PBS twice, re-suspended in 25 ml of 50% ethanol/PBS, and stored at -20 °C. The resulting 30mL suspension was then split, 10 mL remaining in 50% ethanol/PBS and the remaining 20 mL re-suspended in 100% ethanol.

JKHS: 20 ml of the sediment was extracted and the top of syringes was sealed by parafilm. Subsequently, the syringes were put into plastic bags, flushed with nitrogen, sealed, and stored at 4 °C.

(2) WRC sampling

The standard procedure (see, **Table 6.3.1**) of WRC sampling in this expedition was: 1) cut WRC: 2) place it in impermeable plastic bag: 3) flush with nitrogen gas: 4) vacuum seal: 5) store at 4°C or -80°C.

For IW WRCs, the vacuum seal was omitted. Before each cutting, the scraper was washed in Milli-Q water and wiped to avoid crossover contamination between samples. 70% ethanol was used to clean the scraper for microbiological samples whereas it was never used for IW samples because it might have interfered with downstream analyses.

The exceptions other than written above were described below. Sampling reports for all cores are shown in **Appendix 1**.

- **FSWR:** WRC was vertically cut into 2 sections before nitrogen flush and vacuum seal.
- YMWR1: 50-cm WRC was cut into 5 of 10 cm sections before nitrogen flush and vacuum seal.

THWR: WRC was put into aluminum bag and stored at -80 °C.

NXWR: WRC was stored at -80 °C.

NAME	CODE	Sample volume (cm)		How to sampleing	STORAGE
Akira ljiri	906AIWR	10	1/core	Flash with N ₂ / Vacuum sealed	4 °C
Frauke Schmidt	906FSWR	20	1/core	Split in two vertically, then flash with N_2 /vacuum sealed seperately	4°C
Gail Arnold	906GAWR	5	1/core	Flash with N ₂ / Vacuum sealed	4°C
Jens Kallmeyer	906JKWR1	10	1/core	Flash with N ₂ / sealed	4°C
Yuki Morono	906YMWR1	50	1/core	Cut into $5x10$ cm WRCs, then flash with $N_2/Vacuum$ sealed seperately	4°C
Sanae Sakai	906SSWR	10	1/core	Flash with N ₂ /Vacuum sealed	4°C
Tatsuhiko Hoshino	906THWR	30	1/core	Place in aluminum bag	-80°C
Nan Xiao	RMS	10	1/core	Place in aluminum bag	-80°C
	906NXWR	10	1 core	Place in plastic bag	4°C
Hitoshi Tomaru	906HTIWWR	20	1/core	Purge with N2 and seal, NOT Vacuum, take it downstairs	
Frauke Schmidt	906FSIWWR	40	1/core	Purge with N2 and seal, NOT Vacuum, take it downstairs 4°C	
Gail Arnold	906GAIWWR	50	2/core	Purge with N2 and seal, NOT Vacuum, take it downstairs	

 Table 6.3.1.
 Sampling procedure for whole round core.



Fig. 6.3.1. Procedure for syringe sampling on the core cutting area.

6.4. Direct measurements of turnover rates

reported by Jens Kallmeyer and Rishi Ram Adhikari

6.4.1. Sample processing

For sulfate reduction rate measurements samples were taken from a single 10 cm WRC from each HPCS core. Glass barrels equipped with a plunger from a 5 cc disposable syringe were used to retrieve small sub-cores with an intact sedimentary structure. Due to massive degassing of the cores and the resulting voids, the sedimentary structure was not fully intact. Also mud clasts and presumably carbonate concretions made sampling of intact sedimentary structures rather difficult.

From each WRC a total of 10 sub-cores with a volume of about 4 cc were taken. Each sample was incubated with 15 μ l of Na₂³⁵SO₄ solution, activity ca. 1.85 MBq. The samples were split as follows:

Sample 1-3: Just with radiotracer, incubated at atmospheric pressure

Sample 4-6: with an additional injection of 20 µl of 0.1 M Na-acetate, incubated at atmospheric pressure

Sample 7-9: incubated at ca. 20 MPa pressure, with a syringe containing 3 ml of Methane

Sample 10: Time Zero blank, fixed about 10 minutes after injection of radiotracer.

Samples 1-9 were incubated for 24 hours at 4° C in the dark. Incubation was stopped by transferring the sediment into 20 ml of 20% (w/v) Zinc acetate solution, followed by immediate freezing to stop all microbial activity and to precipitate all hydrogen sulfide. After freezing for about 12 hours the samples were thawed and will be transported to the home laboratory for analysis.

Due to legal issues the planned Tritium-Hydrogenase assay could not be performed on board. Samples were preserved both at +4°C and at -20°C for later analysis in the home laboratory.

6.5. Interstitial water sampling and shipboard analyses

reported by Hitoshi Tomaru, Hnas Røy, Gail Arnold, Manabu Nishizawa, Frauke Schmidt, Atsushi Tani

6.5.1. Introduction

Structure and material within the mud volcano system has been supported by interstitial water transport. Source and migration history of interstitial waters are essential for describing how the mud volcano has been developed and contributed to the mass transport from the deep to the shallow interval in mud volcano. Furthermore, interstitial water is a medium for microbial activities and hence geochemical properties are important factor to constrain the activities in the unique locally active geological setting. Inorganic/organic geochemical analyses proposed for Expedition 906 provide key date sets that disclose the nature of mud-volcano system.

6.5.2. Whole round core sampling

The interstitial water samples for general shipboard analyses were obtained generally from ~30 cm long whole round cores (WRCs) from the bottom of all sections. These whole round samples taken to the laboratory for immediate X-ray CT (X-CT) scanning and sectioned and sealed in a nitrogen-filled plastic bag in the sampling room, and stored in a 4 °C refrigerator until squeezing.

After extraction from the core liner, the surface of each WRC was carefully scraped to avoid sediments that had potentially been contaminated from seawater, drilling fluid, oxidation, and smearing in the borehole. The clean inner parts of the core were then placed into a Manheim-type titanium squeezer and squeezed with a laboratory hydraulic press at the gauge pressures up to 25,000 lbs. Interstitial water was passed through a filter paper fitted on a stainless steel screens at the bottom of the squeezer. Fluids from the squeezing process passed through a 0.45 µM disposable filter into an acid-washed 25 mL plastic syringe. Interstitial water was sub-sampled for standard shipboard analyses (salinity, alkalinity, pmH, concentrations of Br, SO₄, PO₄, NH₄, and Cl). Subsamples were collected also for shore-based multi-elemental/isotopic

analyses on the requests by scientists and were stored in glass or plastic vials prepared by each scientist. In addition, individual WRCs were collected for specific analyses that require greater volume of interstitial water (i.e., sample code: 906FSIW, 906GAIW, and 906HTIW2). These samples were also obtained following the IW sampling method described above. Detailed sub-sampling plan is described in **Appendix 2**.

6.5.3. interstitial water analysis

Interstitial water samples were routinely analyzed for salinity as total dissolved solutes with ~100 µL subsample using RX-5000a refractometer (Atago) and for pmH and alkalinity with 3 mL subsample by Gran titration using a Metrohm autotitrator immediately after interstitial water was extracted. The chloride concentration was measured on a 100 µL subsample by titration using silver nitrate in a 0.2 M sodium nitrate solution with the relative standard deviation of 0.5%. Sulfate and bromide concentrations were measured by ICS-1500 ion chromatography (Dionex) using subsamples that were diluted by 1:100 with 18 M Ω Millipore water. The relative standard deviations were 1.1% and 0.4%, respectively. Dissolved phosphate concentration was measured by colorimetric methods with the aid of an UV-2550 UV-Visible spectrophotometer (Shimadzu) at the absorbance of 885 nm with a reproductively better than 1.87%. Because the phosphate concentrations vary, appropriate aliquot of sample or standard solution (100 μ L or 300 μ L) was diluted to the required volume for the analysis with 18 MQ Millipore water in a plastic tube. The mixed solution (ammonium molybdate, sulfuric acid, ascorbic acid, and potassium antimonyl tartrate) was added in the tube and was well mixed, the tube was capped and kept at room temperature to develop color. Dissolved ammonium concentration was also determined with UV-2550 UV-Visible spectrophotometer at the absorbance of 640 nm with a reproductively better than 1.4%. A 0.1 mL of sample aliquot was diluted with 18 MΩ Millipore water, phenol ethanol, sodium nitroprusside, and sodium hypochlorite solutions in the capped plastic tube, and was kept in the dark at room temperature for more than 3 hours to develop colors.

6.5.4. Results

Figure 6.5.1 shows a summary of shipboard geochemical analyses of interstitial waters. All the profiles generally show constant downward trend, and relatively low concentrations compared with general values in the interstitial waters collected from the Nankai Trough area (e.g. IODP NanTroSEIZE expeditions). In addition, contribution of seawater in the samples near the seafloor is negligible because concentrations in the interstitial waters are distinctly low compared to the seawater for CI, NH₄, Br, and SO₄. Contrary to these results, interstitial water value is high for alkalinity resulting from input of allochthonous fluids from the depth reaches the seafloor.

A sample at ~50 mbsf (4H-1) showed values somewhat close to the seawater, indicating seawater (drilling fluid) contamination during core retrieval. Contamination of this section was also confirmed by microscopic analysis of the sediment, revealing small pieces of "carpet" material, likely introduced during core recovery.

Gas hydrates are potentially contained in Sections 6H-4 and 8H-5 (indicated by blue line in **Fig. 6.5.1**) because low temperature anomalies were observed by infrared camera on the core cutting area. Concentrations of dissolved ions are, however, slightly lower than the adjoining samples, amount of gas hydrates are likely small: e.g. section contained very thin layered or small flake-like gas hydrate.

6.5.5. Shore-based research plans

Akira Ijiri: 906AllW1-3

Dissolved inorganic carbon (DIC), methane & ethane concentration in interstitial waters, d¹⁸O, and d¹³C acetate will be measured to characterize and quantify microbial activities associated with methanogenic processes in deep sediments.

Tomohiro Toki: 906TTIW

B and Li isotopes and concentrations of Na, Ca, K, Mg, Sr, Ba, B, Li, Si, and REE will be measured to examine influence of deep-sourced fluid B and Li that are temperature sensitive elements.

Frauke Schmidt: 906FSIW

Dissolved organic matter (DOM) concentration will be measured to investigate the sources, preservation potential and susceptibility to remineralisation by microbial turnover.

Atsushi Tani: 906ATIW

Alcohols, aldehydes, and possibly amines in sediment, pore water, and methane hydrate will be measured to reveal distribution of C1 and C2 compounds in deep-sea environment sediment.

Gail Arnold: 906GAIW & 906GAIWS

Concentration and isotopic composition of dissolved and solid sulfur species as well as Fe-speciation in the solid phase fraction and the $d^{18}O_{water}$ will be measured to assess the pathways of sulfur cycling (sulfate reduction and sulfide/sulfur oxidative processes) in a mud volcano matrix, either at a macro-scale - where sulfate concentrations exceed 1mM, or at a "cryptic scale" – where sulfate concentrations are between 0.1 and 1.0 mM.

Hans Røy: 906HRIW1-3

Fe(II), sulfide, and sulfate concentrations will be measured to constrain the potential substrates for microbial energy metabolisem within the mud volcano and to quantify as many pools of reactive iron and sulfur species as possible and build a mass balance to detect changes within these pools as function of depth below seafloor.

Hans Røy: 906IWS

Fe(II) and Fe(III)-compounds in the solid phase will be measured to separate and quantify the pools of FeS, FeS₂, FeOOH. A similar, though less detailed, speciation balance will be calculated for Mn species.

Manabu Nishizawa: 906NMIW

Compositional and isotopic distribution of inorganic nitrogen compounds

 $(N_2, N_2O, NH_4^+, NO_2^-, NO_3^-)$ will be measured to understand nitrogen (bio)geodynamics in deep sediments thorough IW and gas samples.

Hitoshi Tomaru: 906HTIW1-2

Concentration and isotopic composition of long-lived radio isotope (¹²⁹I) of dissolved iodine will be measured to determine the age of source organic matter for methane, representing potentially the relative age of fluid that transport within the mud volcano system.



Figure 6.5.1. Depth profile of shipboard geochemical analyses of interstitial waters collected from Site C9004 Hole B.

6.6. Head space gas analysis

reported by Akira Ijiri and Tomohiro Toki

Head space H₂

A 2.5 cm³ of sediment was sub-sampled by cut-off plastic syringe. The syringe was immediately capped by silicone rubber stopper and brought to laboratory. The syringe-containing sample was extruded into 5 mL of glass vial in which 1 mL of saturated sodium chloride solution in distilled, deionized water (DDW) had been supplemented to make slurry. After the vial capped with a Teflon-coated butyl rubber stopper, the vial was shaken and stored for 30 min at room temperature. The 2–3 mL of gas phase in the vial was taken by a gas tight syringe and then manually injected into the SRI GC equipped with a reduction gas detector (RGD) to determine H₂. The GC with RGD was brought by JAMSTEC Kochi Institute for Core Sample Research (KCC) as 3rd party tool. Chromatographic response on the GC was calibrated against the introduced volume of prepared standard gas (104 ppm of H₂).

Headspace methane

A 2.5 cm³ of sediment was sub-sampled by cut-off plastic syringe at the same point as H_2 measurement sampling. The syringe-containing sample was immediately extruded into 10 mL of glass vial in which 1 mL of saturated sodium chloride solution in distilled, deionized water (DDW) had been supplemented to make slurry. The sample was brought to KCC for on shore measurement of concentration and stable isotopic compositions of hydrocarbon gases. The raw data are shown in **Table 6.6.1**.

3rd party Summary Sheet									
Exp:	906	Ins	strument:	GC-	RD Samp		ple Code:	906A	HS2
		-				-			
							S	Set	
							Para	meter	
					Interva		Depth		ЦЭ
Site	Hole	Core	Туре	Section	Тор	Bottom	Тор	Bottom	ΠZ
					(cm)	(cm)	CSF-B [m]	CSF-B [m]	ppm
C9004	В	1	Н	5	0	4			8.8
C9004	В	1	Н	6	0	4			7.2
C9004	В	1	Н	8	0	4			3 <u>.</u> 1
C9004	В	2	H	2	0	4			6.7
C9004	B	2	H	3	0	4			8.6
C9004	В	2	H	4	0	4			4.5
C9004	В	2	H	6	0	4			6.4
C9004	В	2	H	7	0	4			6.1
C9004	В	3	Н	2B					19.9
C9004	B	3	H	<u>3B</u>					6.5
C9004	B	3	H	<u>4B</u>					9.9
C9004	В	3	H	5B					9.1
C9004	B	3	H	<u>6B</u>					147.3
C9004	В	6	H	<u>4B</u>					0.4
<u>C9004</u>	В	6	H	<u>5B</u>					0.4
<u>C9004</u>	B	6	H	<u>6B</u>					0.6
<u>C9004</u>	В	8	H	3	0	4			2.2
<u>C9004</u>	В	8	H	4	0	4			0.8
<u>C9004</u>	В	8	H	<u>4B</u>	0				3.1
C9004	В	8	H	6	0	4			3.5
C9004		5	P						9.0
C9004		5 7					_		5.5
<u>C9004</u>		1							145.9
C9004		1							56.4
C9004		1	D						50.4
C9004		1	P						123.0
00004	U								120.0

Table 6.6.1. Head space analysis of hydrogen concentration.

6.7. Pressure coring and PCATS (Pressure Core Analysis and Transfer System)

reported by Yuki Morono, Hidenori Kumagai, Tomohiro Toki, Wataru Tanikawa

6.7.1. Introduction

"Pressure core" is the cored sample taken from geological formations with keeping *in-situ* pressure during recovery. It is important to maintain physical, geochemical and biological characteristics under the *in-situ* pressure, especially for gas hydrate-bearing sediment. During Expedition 906, using a newly developed 3.5 meters-pressure coring system (PCS), so called "Hybrid-PCS", we attempted to retrieve pressure cores of methane hydrate-bearing sediment from the mud-volcano no. 5 at the water depth of approximately 2000 meters (i.e., *in situ* pressure is ~200 bar). Decreasing pressure and increasing temperature during core recovery from the seabed is critical for maintain hydrate structure and volatile gas because of the hydrate dissociation. The dissociation of gas hydrate creates large volumes of gas, which may expand and destroy the complete fabric of core prior to it reaching the surface.

6.7.2. Coring and transfer of *in-situ* pressure core

All *in-situ* pressure cores were obtained by a newly developed Hybrid Pressure Coring System (Hybrid-PCS). Expedition 906 is the first operation of Hybrid-PCS that has been customized for the use of *Chikyu*. The core length of Hybrid-PCS is 3.5 meter. During Expedition 906, we tested two core barrels: i.e., punching shoe and cutting shoe. Temperature and pressure logger was deployed on the coring system, which logs are shown in **Appendix 5**.

After pressure core recovery, the core barrel was placed in a vertical ice shock-chamber on the rig floor at least for ~30 min to keep the low pressure to avoid hydrate dissociation. Then, the core barrel was placed in another ice pool horizontally and the autoclave part was detached. The core chamber was then immediately transferred to GeoTek PCATS container lab by crane and connected to the transfer system (**Fig. 6.7.1**, **Table 6.7.1**). After the connection

of the core chamber to PCATS, the inside core was transferred to another aluminum high-pressure chamber without depressurization. The core section can be checked by X-ray during the transfer operation (**Fig. 6.7.2**).

Hole	Core	Туре	Тор	Bottom	Recovery
В	3	P/T	1952.5	1955.5	0.15 (1.7%)
В	5	P/T	1988.0	1991.0	0.7
В	7	P/X	2038.0	2042.0	0.2
В	9	P/X	2118.5	2122.5	0.0
С	1	Р	0	6.5	0.9

 Table 6.6.1.
 Summary of hybrid-PCS core recovery.



Fig. 6.7.1. Transfer of pressure core into GeoTek PCATS container.



Fig. 6.7.2. X-ray observation of the pressure core.

6.7.3. Gas sampling from the Hybrid-PCS at/after the hydrate production test.

Outline of gas production test

The test is carried out after the X-ray investigation inside of the pressure-vessel. Up to a 30 cm long section can be stored into the dedicated (small) pressure vessel. In standard protocol, pressure is released gently down to just beneath the stability field of the hydrate (~40 bar), in approx. 30 min. In case of the vessel contains any gas hydrate, pressure rebounds toward the hydrate stability field; the pressure became stable at the pressure predicted from the chamber (~ van-inside) temperature, e.g. ~40 bar under 10 °C of the test-van, when sufficient amount of the hydrate is contained. Once reach the stable pressure, the valve from the chamber is opened to introduce pressured gas and/or water into the graduated cylinder. Prior to the test, the cylinder has been filled by water, and any air bubble was carefully removed and water level was adjusted. This depressurize process are repeated while the gas is released.

Gas Sampling

(A) Rn and noble gases

reported by Hidenori Kumagai

On the top of the cylinder, a luer lock connecter was equipped with a stopcock. For on-shore noble gas measurement, approx. 110 mL of the gas was collected directly from the cock into the vacuumed IsoTube.

From the remnant of the gas in the cylinder, up to 800 mL of the gas aliquot is pumped into the Rn measurement apparatus, AlphaGuard PQ2000 Pro, Saphymo GmbH, Germany. The apparatus counts any decay in its ion-chamber as the Rn decay, thus complete filtering any dust from the gas introduced in the chamber is required, which potentially makes memorized contamination. The configuration of the connection at this sampling is shown in **Fig. 6.7.3**.

At the on-line measurement of Rn, maximum Rn concentration >100 Bq/m³ was recorded. This means the obtained hydrate contains some significant amount of the Rn prior to the entire decay (ca. 38 dy as 10 times of the half-life). Although the Rn concentration of the surrounding formation is unknown, this

suggests that the hydrate is retrieved very quickly since its formation.

(B) Gas composition analysis of high-pressure core

reported by Akira Ijiri and Tomohiro Toki

PCAT high-pressure core provides an ideal opportunity to study in-situ gas analyses, which has not been effected by degassing during core recovery. During Expedition 906, the gas was extracted directly from the pressurized chamber through the vacuum line, and then measured pressure of the sample bottle. Using the gas sample, hydrogen concentration was measured as described previously, and the initial data are shown in **Table 6.6.1.** We also performed gas-production test from 10-15 cm high-pressure WRC (see, **Figs 6.7.6-6.7.9**).



Fig. 6.7.3. Schematic diagram of the configuration of Rn and noble gas sampling.

6.7.4. Physical property test using PCAT triaxial

reported by Wataru Tanikawa

Pressure cores are transferred from PCATS to the tri-axial deformation apparatus (PCATS triaxial, **Fig. 6.7.4**) by preserving *in-situ* pressure condition. Sample is covered by rubber jacket during transferring core sample into triaxial testing pressure vessel; therefore, confining pressure can be applied during physical property measurement. Several physical properties, such as permeability, elastic property, and shear strength, are measured using this high-pressure apparatus. Permeability is measured by the steady state flow method. In this method, differential fluid pressure is applied between both ends of the specimen by keeping constant flow rate using two flow pumps. Permeability is then calculated by differential pressure and flow rate. The methodology for elastic properties (shear modulus and Young's modulus) of sediment is described by Priest et al. (2005, 2009). Low axial strain and shear strain is applied in this method, therefore no permanent deformation is achieved. A common triaxial deformation test is also performed by applying large axial strain to core samples, and strength of sediment is measured.

6.6.5. References

- Priest, J.A., Best, A. I., and Clayton, C. R. I., 2005, A laboratory investigation into the seismic velocities of methane gas hydrate-bearing sand, *J. Geophys. Res.*, **110**, B04102, doi:10.1029/2004JB003259
- Priest, J. A., Rees, E. V. L. and Clayton, C. R. I., 2009, Influence of gas hydrate morphology on the seismic velocities of sands, *J. Geophys. Res.*, **114**, B11205, doi:10.1029/2009JB006284



Fig. 6.7.4. PCATS triaxial system.



Fig. 6.7.5. Vertical profiles of gamma-ray density, P-wave velocity and X-ray CT image of Core C9004C-1P.



 Table 6.7.6. Gas production test of C9004B-5P-2.



Fig. 6.7.8. Gas production test of C9004C-1P-6.



Fig. 6.7.9. Gas production test of C9004C-1PCC.

Appendix 1. Syringe and WRC sampling Note.

Site C9004 Hole B Core 1 Type H Time on deck: June 25 2012 22:52 Samplers: Hans Røy & David Case Number of sections: 8

General. Top of core with very little mud, distributed as small chunks on the liner walls. Sampling slow. Section one skipped. Most samples in section two and three were not possible to take with good aseptic technique. All compromised samples have been noted as compromised in the chart. Samples with no comments are good. Cut faces had to be sampled in both side of the cut in all but the last cut (section 7-8). Section 7 was short and was not sampled because there was a good long section 8. Section 8 was sampled in top only.

Detail.

Unless otherwise noted, sampling volumes were reasonably close to:

ATHS: 3cc DCHS1: 3cc DCHS2: 5cc AIHS1: 2.5cc AIHS2: 2.5cc YMHS: 5cc JKHS1: 20cc

Section 1. This section was skipped entirely by scientist sampling, due to a lack of available mud. The D/V Chikyu technicians took one safety gas measurement from the bottom of this section.

Section 2. Due to a low abundance of mud in this section, aseptic technique was not possible. Samples taken in this section were:

- ATHS, bottom of section. Non-sterile.
- DCHS1, bottom of section. Non-sterile.
- DCHS2, bottom of section. Non-sterile.

Section 3. Due to a low abundance of mud in this section, aseptic technique was not possible. Mud was chunky. Samples taken in this section were:

- ATHS, bottom of section. Non-sterile.
- AIHS1, top of section. Non-sterile.
- AIHS2, top of section. Non-sterile. Less than 2.5cc.
- YMHS, top of section. Non-sterile. Time of sample mixing with PFA: 00:12 (June 26)

Section 4. Here the abundance of mud increased enough to allow proper sterile technique in some samples. Section ends were increasingly filled. Mud retained chunks but increasingly smooth and soft. Samples taken in this section were:

- ATHS, bottom of section. 2cc.
- DCHS1, top of section.
- DCHS1, bottom of section.
- DCHS2, top of section. 3cc.
- DCHS2, bottom of section. 3cc.
- AIHS1, top of section. Non-sterile.
- AIHS1, bottom of section.
- AIHS2, top of section. Non-sterile. 2cc.
- YMHS, top of section. Time of sample mixing with PFA: 00:30?(June 25)

Section 5. Here the mud abundance was sufficient to maintain aseptic technique whenever required (samples sterile unless otherwise noted). Mud was smooth and soft. Samples taken in this section were:

• ATHS, bottom of section

• DCHS2, bottom of section. Tried sampling top of Sec. 6, but failed and so sampled bottom of Sec. 5. 3cc.

- AIHS2, top of section
- YMHS, top of section. Time of sample mixing with PFA: 00:40 (June 25)
- JKHS1, bottom of section

Section 6. High mud abundance, good quality sampling. Samples taken were:

- DCHS1, top of section. Sample was adjacent to CH₄ hydrate.
- AIHS1, top of section. 1.5cc
- AIHS2, top of section. 2cc.
- YMHS, top of section. Time of sample mixing with PFA: 00:55 (June 25)

Section 7. This section was short and was therefore not sampled. The boundaries of section seven were defined by two void spaces in the core.

Section 8. High quality end to this core, a full suite of all samples was able to be taken. Samples retrieved were:

- ATHS, top of section. Procedure error: sample was plugged with rubber plug.
- DCHS1, top of section.
- DCHS2, top of section. 3cc.
- AIHS1, top of section.

• AIHS2, top of section. Procedure error: sample end was covered with parafilm and sample was placed in plastic baggie.

- YMHS, top of section. Time of sample mixing with PFA: 01:20 (June 25)
- JKHS1, top of section.

WRC sampling

Each WRC was taken and stored according to the table of sampling procedure unless otherwise noticed.

Section 1. No WRC was taken because of low recovery <10% and abundance of breccias

Section 2. No WRC was taken because of low recovery <5% and abundance of breccias

Section 3. No WRC was taken because of low recovery <5% and abundance of breccias

Section 4. No WRC was taken because of low recovery <20% and abundance of breccias

Section 5. Middle parts of the section had low recovery <20% and was not taken for WRCs. 0-40 cm: IW 100-140 cm: IW

Section6. The length is 71 cm with recovery of 60% 0-71 cm (whole section): GAIW

Section7. The length is 113.5 0-50 cm: FSIW 100-113.5 cm: RMS Exp. 906 <u>Site C9004</u> <u>Hole</u> <u>Core 2</u> <u>Type H</u> <u>Time on deck: June 25 2012 22:52</u> <u>Samplers: Tatsuhiko Hoshino, Sanae Sakai, Rishi Adhikari, and Yuki Morono</u> <u>Number of sections: 7</u>

General. The core contained large void in the middle. Total recovery was 7m. The first section was skipped.

All syringe samples except for JKHS (Jens samples) were taken from the top sides. Tomaru-san observed the temperature of core liner but did not found abnormal low temperature indicating existence of methane hydrate. After the sectioning, no methane hydrate was found. Cores contained much breccia, some parts of cores were not suitable for WRC sampling while core recovery was high.

Syringe sampling

Detail.

For all sampling volumes were reasonably close to:

ATHS: 3cc DCHS1: 3cc DCHS2: 5cc AIHS1: 2.5cc AIHS2: 2.5cc YMHS: 5cc JKHS1: 20cc

Section 1. This section was skipped entirely by scientist sampling, due to a lack of available mud. Safety gas measurement sample was taken from the bottom of this section.

Section 2. Mud was very soft and sticky:

- ATHS, top of section. Non-sterile.
- DCHS1, top of section. Non-sterile.
- DCHS2, top of section. Non-sterile.
- AIHS1, top of section. Non-sterile
- AIHS2, top of section. Non-sterile
- YMHS, top of section. sterile, at 0400

Section 3. Mud got harder than the top sections.

- ATHS, top of section. Non-sterile.
- DCHS1, top of section. Non-sterile.
- DCHS2, top of section. Non-sterile.
- AIHS1, top of section. Non-sterile
- AIHS2, top of section. Non-sterile
- YMHS, top of section. sterile, at 0410
- JKHS, bottom of section. sterile

Section 4. Mud was hard. Inserting syringe sampler was difficult especially for large-volume syringes such as 20 ml and 10 ml.

• ATHS, top of section. Non-sterile.

• DCHS1, top of section. Non-sterile.

- DCHS2, top of section. Non-sterile.
- AIHS1, top of section. Non-sterile
- AIHS2, top of section. Non-sterile
- YMHS, top of section. sterile, at 0420

Section 5. Mud was hard. Inserting syringe sampler was difficult especially for large-volume syringes such as 20 ml and 10 ml.

- ATHS, top of section. Non-sterile.
- DCHS1, top of section. Non-sterile.
- DCHS2, top of section. Non-sterile.
- AIHS1, top of section. Non-sterile
- AIHS2, top of section. Non-sterile
- YMHS, top of section. sterile, at 0425

Section 6. Mud was hard. Inserting syringe sampler was difficult especially for large-volume syringes such as 20 ml and 10 ml.

- ATHS, top of section. Non-sterile.
 - DCHS1, top of section. Non-sterile.
 - DCHS2, top of section. Non-sterile.
 - AIHS1, top of section. Non-sterile
 - AIHS2, top of section. Non-sterile
 - YMHS, top of section. sterile, at 0426

Section 7. Mud was hard. Inserting syringe sampler was difficult especially for large-volume syringes such as 20 ml and 10 ml.

- ATHS, top of section. Non-sterile.
- DCHS1, top of section. Non-sterile.
- DCHS2, top of section. Non-sterile.
- AIHS1, top of section. Non-sterile
- AIHS2, top of section. Non-sterile
- YMHS, top of section. sterile, at 0430

WRC sampling

WRCs for IW (sections 2, 5, and 6) were taken at first. Each WRC was taken and stored according to the table of sampling procedure unless otherwise noticed.

Detail

Section 1. Skipped due to the very short length

Section 2.

0-50 cm: IW 50-100 cm: GAIW 100-140 cm: FSIW

Section 3.

0-20 cm: THWR, N2 flash, vacume sealed-> -80°C 20-30 cm : GAWR, 30-40 cm: JKWR, N2 flash, NOT valucumed, and sealed -> 4°C 40-50 cm: SSWR 50-60 cm: AIWR 60-70 cm: FSWR 70-132 cm: YMWR1 ->cut into 10-cm sections for first 5: last one, 13 cm WRC

Section 4. No WRC was taken due to breccia

Section 5.

0-30 cm: IWWR (50 cm WRC was requested, but it was shortened to 30 cm)

Section 6. 17-27 cm: RMS, put in an aluminum bag -> -80°C 60-110 cm: GAIW 110-141 cm: IWWR

Section 7 42-56 cm: NX Exp. 906 <u>Site C9004</u> <u>Hole B</u> <u>Core 4</u> <u>Type H</u> <u>Time on deck: June 26 2012 11:45</u> <u>Samplers: David Case, Atsushi Tani & Takeshi Terada</u> <u>Number of sections: 7</u>

General. Core was generally of high quality, with smooth sediment which worked well with syringe samples. David inserted syringes, followed by Terada-san extracting syringes and Tani-san keeping track of labels and notes. In the manner the samples worked sequentially downcore. The top (section 1) and bottom (sections 6, 7) of core had void spaces but the middle of the core (sections 2 through 5) had few voids and the sediment was smooth. Sampling was performed over the course of ~30 minutes. Infrared imaging did not reveal any notable abnormalities.

Detail.

Unless otherwise noted, sampling volumes were reasonably close to:

ATHS: 3cc DCHS1: 3cc DCHS2: 5cc AIHS1: 2.5cc AIHS2: 2.5cc YMHS: 5cc JKHS1: 20cc

Section 1. This section was skipped entirely by scientist sampling, due to a lack of available mud.

Section 2. The top of section 2 still had void space varying over from section 1, so sampling was done at the bottom of section 2. Here there was good sediment quantity and quality for syringe sampling. The samples taken here were:

- ATHS, bottom of section.
- DCHS1, bottom of section.
- DCHS2, bottom of section.
- AIHS1, bottom of section.
- AIHS2, bottom of section.
- YMHS, bottom of section. Time of sample mixing with PFA: 12:47 (June 26)

Section 3. Sediment quality and quantity were both high in this section. Samples could have been taken from the top of section three, but for intra-core consistency all samples were taken from the bottom of section three. Samples taken were:

- ATHS, bottom of section.
- DCHS1, bottom of section.
- DCHS2, bottom of section.
- AIHS1, bottom of section.
- AIHS2, bottom of section.
- YMHS, bottom of section. Time of sample mixing with PFA: 12:54 (June 26)

Section 4. Once again, sediment quality and quantity were overall high in this section. There began to be slight difficulty inserting syringes, due to what felt like chunks impeding the syringe upon entry. This was not a large issue, however, and the syringe rarely had to be wiggled to gain entry. All sampling was still able to be performed robustly.

- ATHS, bottom of section.
- DCHS1, bottom of section.
- DCHS2, bottom of section.

- AIHS1, bottom of section.
- AIHS2, bottom of section.
- YMHS, bottom of section. Time of sample mixing with PFA: 13:01 (June 26)

Section 5. Sediment quality was high, with enough quantity for a full sample set. Difficulty of syringe entry continued to increase, though sediment resistance and/or chunks still did not prevent robust sampling. Due to the approaching end of the core, and the increasing difficulty in syringe entry, David decided to acquire the one/core Jens Kallmeyer syringe sample. Because the regular 6 samples filled up the bottom of section 5, the JKHS1 sample was taken from the top of section 5. It thus best correlates with data from samples at the bottom of section 4.

- ATHS, bottom of section.
 - DCHS1, bottom of section.
 - DCHS2, bottom of section.
 - AIHS1, bottom of section.
 - AIHS2, bottom of section.
 - YMHS, bottom of section. Time of sample mixing with PFA: 13:13 (June 26)
 - JKHS1, top of section. Only 18cc (of 20cc requested) were obtained.

Section 6. Here mud quantity became low, such that taking all standard samples from the bottom of the section was not feasible. Mud quality also became chunky, such that syringe injection was difficult and required jiggling. Only two samples were able to be taken from section six, both from the bottom:

- DCHS1, bottom of section. Low quality sample (required jiggling syringe)
- DCHS2, bottom of section. Low quality sample (required jiggling syringe)

Section 7. This section had many void spaces and the sediment quality was not high. Four samples were taken from the top of the section:

- ATHS, top of section.
- AIHS1, top of section.
- AIHS2, top of section. Low quality sample, much syringe jiggling required.
- YMHS, top of section. Time of sample mixing with PFA: 13:19 (June 26)

WRC sampling

All sections were cut sequentially from core 1 to 7. Each WRC was taken and stored according to the table of sampling procedure unless otherwise noticed. Section 4 of the core had been twisted in drill operations and thus had a spiral shape which made WRC cutting difficult and likely impacted the internal sediment distribution in that section.

Detail Section 1. Length 90cm 0-30 cm: IW

Section 2. Length 141cm 20-50 cm: IW

Section 3. Length 130cm 12-50 cm: FSIW 50-100 cm: GAIW 100-131 cm: IW

Section 4. Length 140 cm 0-10 cm: GAWR 10-20 cm: JKWR 20-30 cm: AIWR 30-40 cm: SSWR 40-60 cm: FSWR. Cut in half vertically, individually flushed each half with N2 and sealed under vacuum, stored at 4°C 60-90 cm: THWR 90-140 cm: YHWR1, split into 5x10cm subsamples. **Section 5.** Lenth 140 cm 0-45cm: GAIW 45-75 cm: IW

Section 6. Length 84cm 54-84cm: IW

Section 7. Length 129 cm 35-50 cm: RMS 90-129 cm: YMWR1

Exp. 906 <u>Site C9004</u> <u>Hole B</u> <u>Core 6</u> <u>Type H</u> <u>Time on deck: June 27 2012 3:59</u> <u>Samplers: Tatsuhiko Hoshino, Sanae Sakai, Rishi Adhikari, Yuki Morono and Hitoshi Tomaru</u> Number of sections: 6

General.

Core recovery of very low probably due to explosion of methane hydrate. The core had many void space. Based on IR camera observation, the temperature of almost entire core was lower than previous cores. The lowest temperature was observed at the top of section 4. However, no methane hydrate was observed anywhere in the whole core. It was observed that methane gas bubbles were generated when sediment from the core catcher came to the cutting area. Several pieces of methane hydrate of around 5 mm were also find in the sediment. Sections from 1 to 3 were the explored sediments and not used for syringe sampling. All syringe samples other than JKHS were taken from the bottom of sections 4-6.

Detail.

Unless otherwise noted, sampling volumes were reasonably close to:

ATHS: 3cc DCHS1: 3cc DCHS2: 5cc AIHS1: 2.5cc AIHS2: 2.5cc YMHS: 5cc JKHS1: 20cc

Section 1-3. These cores were explored sediments. This section was skipped entirely by scientist sampling,

Section 4. The pieces of cores were concatenated into one section.

- ATHS, bottom of section.
- DCHS1, bottom of section.
- DCHS2, bottom of section.
- AIHS1, bottom of section.
- AIHS2, bottom of section.
- YMHS, bottom of section.

Section 5.

- ATHS, bottom of section.
- DCHS1, bottom of section.
- DCHS2, bottom of section.
- AIHS1, bottom of section.
- AIHS2, bottom of section.
- YMHS, top of section.
- JKHS, top of section.

Section 6.

- ATHS, bottom of section.
- DCHS1, bottom of section.
- DCHS2, bottom of section.
- AIHS1, bottom of section.
- AIHS2, bottom of section.
- YMHS, top of section.
WRC sampling

Only three sections were available for the WRC sampling.

Detail Section 4. Length 64.5cm 0-33 cm: IW 33-48 cm: AIWR 48-64.5 cm: SSWR

Section 5. Length 56.5cm 0-10 cm: JKWR 30-56.5 cm: THWR

Section 6. Length 93cm 50-73 cm: IW 73-93 cm: FSWR

Exp. 906 Site C9004 Hole B Core 8 Type H Time on deck: June 27 2012 15:07 Samplers: David Case, Takeshi Terada, and Atsushi Tani Number of sections: 8

General.

Core had many void spaces, likely due to methane hydrates. Infrared camera did not detect temperature abnormalities, though. MQJ technicians drilled small holes in core to relieve pressure. This off-gassing caused sediment to shift up and down core somewhat. The sediment smoothness varied between sections but in general the sediment was very hard, and difficult to sample with syringe. In some places a small bubbling was observed coming out of the sediment, likely the dissociation of small methane hydrate chunks. Following sampling, void spaces were removed by pushing the sediment back together.

Detail.

Unless otherwise noted, sampling volumes were reasonably close to:

ATHS: 3cc DCHS1: 3cc DCHS2: 5cc AIHS1: 2.5cc AIHS2: 2.5cc YMHS: 5cc JKHS1: 20cc

Section 1-2. These sections were completely disturbed by hydrate expansion and were not syringe sampled.

Section 3. This section had relatively accessible sediment for syringe sampling, though overall the sediment was hard and difficult to inject syringes into.

- ATHS, top of section.
- DCHS1, top of section.
- DCHS2, top of section.
- AIHS1, top of section.
- AIHS2, top of section.

• YMHS, top of section. PFA fixation begun at: 16:09 July 27 2012

Section 4. Sediment had many sites of small bubbling, indicating the dissolution of methane hydrates. Sampling was of high quality, although sediment was more hard than ideal.

- ATHS, top of section.
- DCHS1, top of section.
- DCHS2, top of section.
- AIHS1, top of section.
- AIHS2, top of section.
- YMHS, top of section. PFA fixation begun at: 16:17 July 27 2012
- JKHS, top of section.
- DCHS1, bottom of section
- AIHS1, bottom of section
- AIHS2, bottom of section

Section 5. Sediment became too hard and difficult to put all syringes in together. Consequently, syringes were split between the top of section 5 and the bottom of section 4.

- ATHS, top of section.
- DCHS2, top of section.

• YMHS, top of section. Not sterile because so much wiggling of syringe had to be injected into multiple locations in order to retrieve enough sediment. PFA fixation begun at: 16:20 July 27 2012

- ATHS, bottom of section
- DCHS1, bottom of section
- DCHS2, bottom of section

Section 6. Sediment amount was low because of void spaces. Quality of sediment present was high. Sampling was split between the top of section 6 and bottom of section 5 because there was not enough sediment for all syringes at the top of section 6.

- AIHS1, top of section
- AIHS2, top of section
- YMHS, top of section. PFA fixation begun at: 16:28 July 27 2012
- JKHS, bottom of section.

Section 7. Sediment quality improved, mud was softer and in higher abundance.

- ATHS, top of section.
 - DCHS1, top of section.
 - DCHS2, top of section.
 - AIHS1, top of section.
 - AIHS2, top of section.
 - YMHS, top of section. PFA fixation begun at: 16:42 July 27 2012

WRC sampling

Detail

Section 1. Length 150cm. Not sampled for WRC, sediment was too disturbed.

Section 2. Length 45cm. Not sampled for WRC, too many voids.

Section 3. Length 109cm 0-50cm: GAIW 50-80cm: IW 80-90cm: JKWR 90-100cm: AIWR 100-109cm: GAWR

Section 4. Length 97.5cm 0-41cm: FSIW

60-80cm: FSWR

Section 5. Length 135.5cm 30-40cm: SSWR 98-116cm: IW 116-135.5cm: IW (Methane hydrate signatures observed here in CT)

Section 6. Length 129.5cm 0-66cm: YMWR 66-100: THWR 100-129.5: IW

Section 7. Length 133cm 0-20cm: FSWR 20-30cm: AIWR 30-40cm: RMS 50-100cm: GAIW 100-133cm: IW

Section 8. Length 92cm. Not sampled for any purpose, too many voids.

Exp. 906 Site C9004 Hole B Core 10 Type X Time on deck: June 28 2012 3:04 Samplers: Tatsuhiko Hoshino, Sanae Sakai, Yuki Morono and Hitoshi Tomaru Number of sections: 1

General.

No core recovery. The ESCS coring system did not seem to be suitable for the sediment. One section was obtained but contains only gravels and muddy water. Therefore, no syringe sample and WRC was taken from this core.

Appendix 2. Interstitial water sampling flow.





Frauke Schmidt

Frauke Schmidt

Frauke Schmidt



Chikyu expedition 905 GAIW work flow v. 2, June 26 2012

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Chikyu expedition 905 IW work flow v. 2, June 26 2012



Hans Ray

Appendix 3. Location of whole round core samples on X-ray CT scan image.





906 Kumano Mud Volcano X-ray CT images

Appendix 4. Sample List.



Expedition906SiteC9004BExpedition nameHybrid PCS Test at Mud Volcano in Kumano-nada

Scientists Sample Summary

Main Requester	Request ID	Sample Code	Purpose	Number of samples
Tomohiro Toki	906-01	906TTIW	Interstitial water sample	15
David Case		906DC	Discrete sample	5
	906-02	906DCHS	Headspace sediment sample	26
Satoru Muraoka		906YN1	Discrete sample	24
	906-03	906YN2	Discrete sample	6
		906GAIWB	Interstitial water sample	8
		906GAIWC	Interstitial water sample	8
1	906–04	906GAIWD	Interstitial water sample	8
		906GAIWF	Interstitial water sample	8
Gail Arnold		906GAIWI	Interstitial water sample	8
		906GAIWS	Squeezed sediment sample	6
		906GAIWT	Squeezed sediment sample	6
		906GAIWWR	Whole round core sample for squeezing	8
		906GAWR	Whole round core sample	4
	906–05	906WT1	Discrete sample	37
Wataru Tanikawa		906WT2	Discrete sample	40
		906WTIWS	Squeezed sediment sample	26
Tatsuhiko Hoshino	906-06	906THWR	Whole round core sample	5
	300 00	906NXWR	Whole round core sample	1
Nan Xiao	906-07	006PMS	Whole round core sample	4
	906-08	906HTH	Discrete sample	1
	300 00	906HTIW1	Interstitial water sample	18
Hitoshi Tomaru	906-09	906HTIW2	Interstitial water sample	3
	300 03		Whole round core sample for squeezing	3
		006ESIW1	Interstitial water sample	4
		006551W2	Interstitial water cample	4
Everylan Selemiat	906-10	900F31W2	Squeezed sediment sample	4
		006ESIW/W/D	Whole round core sample for squeezing	4
		00655W/P	Whole round core sample	6
		9005300	Gas sample	2
			Headspace sediment sample	25
	906-11 906-13		Headspace sediment sample	10
Akira Iiiri		900AII132	Interstitial water cample	15
		900AIIW1	Interstitial water sample	15
		900AIIW2	Interstitial water sample	15
		900AIIW3	Whole round core cample	6
			Interstitial water cample	15
Hans Roy			Interstitial water sample	15
		900HRIW2	Interstitial water sample	15
			Squeezed acdiment comple	17
		9001141005	Disercto comple	17
Yuki Morono	906-15		Whole round core comple	7
I	906-16		Headapage addiment comple	7
		906JKHS1	Headspace sediment sample	0
Jens Kalimeyer			Whele round core comple	5
Sanaa Sakai	006 17		Whole round core sample	5
Sallat Sanal	900-17	SUDSSWR	Gas sample	1
Manabu Nishizawa	906-18	900MINGAS	Las sample	15
			Interstitial water sample	15
Tomas Facakar	006 10		Disersta semple	10
Tomas reseker	900-19	90011		12
	906–20		Headenage adjiment comple	2
ALSUSTI TATI			Interestitial water comple	15
		900ATIW	Interstitial water sample	1 10

906-C9004B_SectionSummary_120629_A4.xlsx

Expedition	906	Site	C9004B
Expedition name	Hybrid PCS Tes	t at Mud Vo l cano	in Kumano-nada

Section Summary

Section	Curated length (m)	Top depth [m CSF-A]	Bottom depth [m CSF-A]	Top depth [m CSF-B]	Bottom depth [m CSF-B]	J-CORES section ID	Curatorial comment 1
C9004B-1H-1	1.425	-5.5	-4.075	-5.5	-4.09	00000000000058450	
C9004B-1H-2	1.39	-4.075	-2.685	-4.09	-2.714	00000000000058550	
C9004B-1H-3	1.405	-2.685	-1.28	-2.714	-1.324	00000000000058650	
C9004B-1H-4	1.4	-1.28	0.12	-1.324	0.062	00000000000058750	
C9004B-1H-5	1.41	0.12	1.53	0.062	1.457	00000000000058850	
C9004B-1H-6	0.71	1.53	2.24	1.457	2.159	00000000000058950	All to WRC
C9004B-1H-7	1.135	2.24	3.375	2.159	3.283	00000000000059050	1
C9004B-1H-8	0.48	3.375	3.855	3.283	3.758	000000000000059150	All to WRC
C9004B-1H-CC	0.245	3.855	4.1	3.758	4	000000000000059250	
C9004B-2H-1	0.51	14.5	15.01	14.5	14.998	000000000000059350	Disturbed
C9004B-2H-2	1.395	15.01	16.405	14.998	16.361	000000000000059450	
C9004B-2H-3	1.33	16.405	17.735	16.361	17.661	000000000000059550	All to WRC
C9004B-2H-4	0.91	17.735	18.645	17.661	18.55	000000000000059650	All to WRC
C9004B-2H-5	1.41	18.645	20.055	18.55	19.928	000000000000059750	
C9004B-2H-6	1 41	20.055	21 465	19 928	21.306	000000000000059850	
C9004B-2H-7	0.56	21 465	22 025	21.306	21.853	000000000000059950	
C9004B-2H-CC	0.00	21.400	22.020	21.000	21.000	000000000000000000000000000000000000000	
211 00	0.000			21.000			
C9004B-3D-1	0.15	24	24.15	24	24.15	000000000000000000000000000000000000000	
C9004B 3F 1	0.15	24	24.13	24	24.13	0000000000002330	
00004D 4U 1	0.005		F0.00F			000000000000000000000000000000000000000	Disturbed
	0.905	50 005	50.905	50,000	50.009	000000000000000000000000000000000000000	
C9004B-4H-2	1.41	50.905	52.315	50.869	52.224	00000000000000000250	
C9004B-4H-3	1.31	52.315	53.625	52.224	53.482	0000000000000000350	
C9004B-4H-4	1.41	53.625	55.035	53.482	54.836	00000000000060450	
C9004B-4H-5	1.405	55.035	56.44	54.836	56.186	000000000000060550	
C9004B-4H-6	0.84	56.44	57.28	56.186	56.993	000000000000060650	
C9004B-4H-7	1.29	57.28	58.57	56.993	58.232	000000000000000000000000000000000000000	
C9004B-4H-CC	0.8	58.57	59.37	58.232	59	000000000000060850	
C9004B-5P-1	0.1	59.5	59.6	59.5	59.6	00000000000062550	Empty
C9004B-5P-2	0.305	59.6	59.905	59.6	59.905	00000000000062650	Production test
							Replaced Liner
C9004B-6H-1	1.405	100	101.405	100	101.312	00000000000060950	floor
C9004B-6H-2	1.405	101.405	102.81	101.312	102.624	000000000000061050	Replaced Liner
C9004B-6H-3	0.405	102.81	103.215	102.624	103.002	000000000000061150	Replaced Liner
C9004B-6H-4	0.645	103.215	103.86	103.002	103.604	000000000000061250	All to WRC
C9004B-6H-5	0.565	103.86	104.425	103.604	104.132	000000000000061350	
C9004B-6H-6	0.93	104 425	105 355	104 132	105	000000000000061450	All to WRC
C9004B-6H-CC	0.54	105 355	105 895	105	105 505	000000000000061550	
C9004B-7P-1	0.02	109.5	109.52	109.5	109.52	000000000000063050	in hag
	0.02		100.02				Production test
C9004B-7P-2	0.18	109.52	109.7	109.52	109.7	00000000000063150	in bag
							Disturbed
C9004B-8H-1	1.5	113.5	115	113.5	114.898	00000000000061750	Replaced Liner
C9004B-8H-2	0.455	115	115.455	114.898	115.322	00000000000061850	
C9004B-8H-3	1.09	115.455	116.545	115.322	116.337	00000000000061950	All to WRC
C9004B-8H-4	0.975	116.545	117.52	116.337	117.246	00000000000062050	
C9004B-8H-5	1.355	117.52	118.875	117.246	118.508	000000000000062150	
C9004B-8H-6	1,295	118.875	120.17	118.508	119.715	00000000000062250	All to WRC
C9004B-8H-7	1.33	120.17	121.5	119.715	120.954	00000000000062350	
							Disturbed
C9004B-8H-8	0.92	121.5	122.42	120.954	121.811	00000000000062450	Replaced Liner
C9004B-8H-CC	0.31	122.42	122.73	121.811	122.1	00000000000061650	
L							
C9004B-10X-1	1.01	194	195.01	194	195.01	00000000000062750	
C9004B-10X-CC	0.255	195.01	195.265	195.01	195.265	00000000000062850	
[1

[Note] Em CSF = depth of Core below Sea Floor in meters Em CSF-A: Distance from sea floor to section within recovered core. This scale allows overlap at core and section boundaries. Em CSF-B: Distance from sea floor to section within recovered core is compressed, if ore recovery > 100%, EFor further details, consult the "IODP Depth Scales Terminology" in the IODP web site (http://www.iodp.org/program-policies/).















906-C9004B-7P







