

Resources assessment of methane hydrates in offshore surround Japan

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Japan Oil, Gas and Metals National Corporation (hereinafter, JOGMEC), as a member of research group for resources assessment of Research Consortium for Methane Hydrate Resources in Japan (MH21), is conducting resources assessment of methane hydrates (hereinafter, MHs) in offshore surround Japan.

The seismic interpretation of migrated profiles of two/three dimensions-seismic surveys acquired by geophysical vessel Shigen that JOGMEC owns, are carried out. MHs are being extracted to search the Bottom Simulating Reflector (herein after, BSR) and assumed sand intervals, which is characterized high-amplitude reflected waves. In addition, based on the knowledge of the MHs are correlated to high-velocity, the comparison between extracted MHs and the high velocity anomalies in the velocity-analysis-profiles run in two-dimensions-seismic-survey lines, or the three-dimensions-seismic-survey area carrying out high-density-velocity analysis. This introduced example is an example of seismic interpretation in the data of three dimensions-seismic-survey area, which have not drilled.

The characteristic in this area shows fold structure, which undulates severalfold. In addition, the gaps of the reflected waves can interpret faults, which does not show the large displacement, are seen. Clear velocity contrast to assume BSR and the high-velocity anomaly above BSR are confirmed in the high-density-velocity-analysis profiles.

Multiple reflector of assumed sand facies is estimated by the results of seismic interpretation in the migrated profiles of three dimensions seismic survey, and each flow shows different sedimentation environment and geological age. In addition, heterogeneity of lithology (different grain size and sand/ mud ratio) is suggested by the variety of amplitude and velocity distribution in the high-density-velocity-analysis profile. In the high-density-velocity-analysis profile, characteristic high velocity anomalies in the sand facies above BSR are visible, but these are estimated MHs because those anomalies are shown in the sand facies above BSR.

As above, even the area has not been drilled, the existence of MHs can be estimated from seismic interpretation of the migrated profile of the seismic survey, and the velocity distribution of the high-density-velocity-analysis profile. And it is expected that these results become useful information for the plan of the future drilling programme. This introduction is the example of the three dimensions seismic survey area; hence, it is a useful information for a programme of three-dimensions-seismic-survey plan by performing similar interpretation in two-dimensions-seismic-survey lines.

Keywords: Methane hydrate, 3-D seismic reflection survey, High density velocity analysis

In-situ stress analysis using the anelastic strain recovery (ASR) method at the first off-shore gas production test site

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To evaluate in-situ stresses by applying the anelastic strain recovery (ASR) method to drilling cores, four cores retrieved from different depths between 265?313 meters below seafloor in a well at the first offshore gas production test site in the eastern Nankai Trough, Japan were utilized to measure anelastic strains onboard the drilling ship. The onsite ASR measurements suggest this depth range in the study area is a normal faulting stress regime dominated by gravitation. In subsequent laboratory experiments, the cores were returned to their in-situ stress states via the K0 consolidation procedure, then elastic and anelastic strains were measured during and after the unloading process to verify the onboard measurements and quantify in-situ stresses. These laboratory calibration experiments verified that the ASR measurements following the stress release could provide reasonable estimates for the artificially applied preload stress.

Keywords: In-situ stress, Anelastic strain recovery method, Unconsolidated formation, K0 consolidation

Grain size analysis of sands by an optical microscopy/digital image method

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Grain size is a fundamental property of earth materials. However, no two of the many techniques yield a consistent measurement of this property; thus, elucidating the relationships among different methods is valuable for understanding what constitutes grain size. This study compares the grain size distributions analyzed by optical microscopy/digital image (Morphologi G3 instrument (Malvern Ltd; the UK)) and those analyzed by laser diffraction. In most of the investigated samples, the size distributions obtained by both methods were very similar. However, a shift toward a coarser grain-size distribution was observed in the optical microscopy analysis of finer sand samples, and the frequency distribution was broadened. The fractions of sand and silt size fractions were also consistent between the two methods, but optical microscopy indicated a smaller clay size fraction than the laser diffraction method. The median particle size (30 – 200 μm) was similar in both methods. The standard deviation was lower in the optical microscopy method than that in the laser diffraction method. We conclude that optical microscopy is a useful technique for determining the grain size distribution. Additionally, we investigated the particle shape and particle size in the experimental fault formed by ring-shearing test. This study is financially supported by METI and Research Consortium for Methane Hydrate Resources in Japan (the MH21 Research Consortium).

Keywords: grain size analysis, digital image method, laser diffraction method, sand, ring-shear test

Gravels Associated with a Massive Gas Hydrate Obtained from HR14 RC1403-7

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Some subseafloor core samples were obtained in the scientific cruise HR14 performed by the JOGMEC-Hakurei in the eastern margin of Japan Sea. Sediments in most of the sites are mainly composed of muddy fine clastics, and can be often found accompanied by a small amount of very fine to medium grained sandy layers, which are usually observed as thin laminations in muddy layers. The gravel layer was observed thicker than 20 cm associated with the massive methane hydrate layer longer than 130 cm in the RC1403 (Sec. 7). Over than 1,100 gravels were obtained, of which major size, minor size, aspect ratio, roundness, sphericity, lithology and color were investigated. These are generally pebble (average 8.29mm). They are composed only of very well-rounded, greyish white to dark grey calcareous mudstone. According to the probability plot of their grain-size distribution, it consists of one segment showing the normal distribution, which may indicate gravel transportations by a high energy flow.

This research is a part of the METI project entitled "FY2014 promoting research and development on methane hydrate."

Keywords: gas hydrate, Japan Sea, gravel

Pore Characteristics and Early Diagenesis of Muddy Sediments below the Sea Floor in the Eastern Margin of Japan Sea

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Fine-grained sediment samples below the seafloor were retrieved by the MD179 in 2010 and the HR14 in 2014 at the Umitaka Spur, Joetsu Channel, Toyama Trough, Japan Basin, Nishi Tsugaru and Okushiri Ridge areas. It is important to clarify the relationship between burial depths and absolute porosities of the argillaceous sediments in relation to early diagenesis. They consist of silt- to clay-grained particles, and they sometimes contain very fine- to medium-grained thin sandy layers. Average porosities of these fine-grained sediments are 50 % in all study areas, which quickly reduce from 60% to less than 50% within 10 meters and gradually decrease to the depth. However, mean pore sizes in the Nishi Tsugaru are around 1000 nm while 100 nm in the other areas, which tend to decrease with depth. It is suggested that repacking of the muddy particles gradually advances by mechanical compaction, which may crucially influence permeability.

They usually contain much opal-A, quartz, feldspar, illite and smectite that do not change definitely with depth. By optical and microscopic observations, diatom tests, foraminifers and framboidal pyrites are commonly observed, and, in particular, the shapes of diatom are usually various, dominantly fragmental and infrequently preserved. It is remarked that physical diagenesis proceeds first due to mechanical compaction, whereas chemical diagenesis advances very slowly in early diagenesis.

This study was performed as a part of the MH21 Research Consortium on methane hydrate in Japan and the METI project entitled "FY2014 promoting research and development on methane hydrate."

Keywords: Japan Sea, gas hydrate, muddy sediment, diagenesis, burial diagenesis

Oxygen and carbon isotope records of foraminifera and depositional age from HR14-RC1408 core, Mogami Trough

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The eustatic sea level changes in the Late Quaternary have strongly affected the hydrography and environment of Japan Sea because of its geographical condition. Stable isotopes of foraminifera have played important roles in previous paleoceanographic studies of the Japan Sea. (Oba *et al.*, 1991; Ishihama *et al.*, 2014). For example during the Last Glacial Maximum (LGM), freshwater input to the Japan Sea has been assumed to cause the large negative excursion in $d^{18}O$ of planktonic foraminifera.

We analyzed oxygen and carbon isotope of both planktonic and benthic foraminifera with 1 core from Mogami Trough (HR14-RC1408: water depth approximately 830 m, core length 48m). Planktonic and benthic foraminifera from the >150 micrometer fraction were hand-picked, reacted with phosphoric acid at 90 degrees C and analyzed by a GV IsoPrime mass spectrometer at the Center for Advanced Marine Core Research (CMCR), Kochi University. HR14-RC1408 core can be correlated Marine Isotope Stage (MIS) 1 to 9 from $d^{18}O$ of planktonic foraminifera. Negative excursions in $d^{18}O$ of planktonic foraminifera and the existence of *Globigerinoides ruber*, *Neogloboquadrina incompta* (dextral) and *Globigerina bulloides* (thin-walled form) in MIS 1, 5e, and 9 suggest the influx of Tsushima Warm Current. The large negative excursions in $d^{18}O$ of planktonic foraminifera implying freshwater input are recognized during the glacial maximum periods in MIS 2 and 6.

This research is a part of METI's project entitled "FY2014 Promoting research and development on methane hydrate". Isotopic analyses were performed under the cooperative research program of Center for Advanced Marine Core Research (CMCR), Kochi University (Accept No. 14A010, 14B008).

Keywords: Japan Sea, planktonic foraminifera, benthic foraminifera, oxygen isotope, carbon isotope, gas hydrate

Late Quaternary occurrences of methane-related species, *Rutherfordoides cornuta*, in the eastern margin of the Japan Sea

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Introduction

Each Benthic foraminiferal species can be adapted to the various environmental changes of the bottom water and the marine sediments. In the Japan Sea, there are characteristic evidences, that the agglutinated assemblages, differenced from the open ocean, exist in the Japan Sea Proper Water and the drastic changes between suboxic and oxic fossil assemblages correspond to the Quaternary paleoenvironment. On the other hands, unique foraminiferal species might live on some methane seep area around Japan Island because the unique colony of the bacteria and macro-benthos form around methane seep. *Rutherfordoides cornuta*, related to high methane gas content of the sediments in the Sagami Bay (Akimoto et al., 1996), lives on plural area from the northwest Pacific but not from the Japan Sea (Matoba and Nakagawa, 2009). Nakagawa et al. (2009) found the distributions of *Rutherfordoides rotundata* (closely related *R. cornuta*) from the last glacial maximum (LGM; 27-17ka) at the Umitaka Spur, the eastern margin of the Japan Sea. These distributions, corresponded to the negative peak signal of benthic foraminiferal carbon isotope (Takeuchi et al., 2007), indicate to dissociations of methane hydrate and active methane seeps by the lowest sea level.

Purpose

We study benthic foraminiferal assemblages and discover the occurrence of *R. cornuta* (not *R. rotundata*) from the late Quaternary sediment around methane seep area in the eastern margin the Japan Sea (off Joetsu, Oki Trough and Mogami Trough). In this presentation, we will introduce a relationship with the particular geological analysis and the hydrate dissociation at each study area.

Sample and age control

Study samples are collected from three sediment cores recovered on the Umitaka Spur (MD179-3304), a west part of the Oki Trough, and a north part of the Mogami Trough. Each sedimentation age is calculated by the ages of ¹⁴C, tephra, stable isotope events, and bottoms of TL layer.

Results

Obvious occurrences of *R. cornuta* are found from three all core sediments, and these sedimentation ages range to 25-28ka, early LGM. At the thick dark layer (TL-2 layer) above these occurrences, rare benthic foraminiferal number indicates an anoxic bottom condition where most benthic foraminifera couldn't live (Oba et al., 1991), nonetheless, *R. cornuta* and *R. rotundata* lived continuously until LGM. And, these occurrences might show the methane seep environments from 28 ka to LGM (Nakagawa et al., 2009). Therefore, active methane seep events might occur by hydrate dissociations at the Umitaka Spur, west Oki Trough and north Mogami Trough at the same time during the LGM.

In addition, we will discuss the relationship with benthic foraminiferal results and methane seep events during the MIS6 and MIS8, lowstand periods. samples.

Acknowledgement

This research was a part of METI's project entitled "FY2014 Promoting research and development on methane hydrate" .

Keywords: fall of sea level, benthic foraminifera, methane hydrate, last glacial maximum, methane seep, *Rutherfordoides*

Paleoenvironmental change elucidated from the total organic carbon concentration and stable isotope from Mogami trough

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The temporal changes in total organic carbon (TOC) concentration for cored sediment off Joetsu city, Japan Sea is very similar to the millennial scale climate change at the northern hemisphere (Urabe et al., 2014). Then, we analyzed the TOC, total nitrogen (TN) concentration and these stable isotope ratio $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values with high temporal resolution for new core RC1408 from Mogami trough, Japan Sea. TOC concentration and $\delta^{13}\text{C}$ value for RC1408 is high during Holocene, and low in Last glacial maxima (LGM).

The high TOC concentration during middle to late Holocene suggests high biological productivity in the sea surface. The contribution of marine organic matter to TOC was increasing with sea level rising, suggesting the decreasing trend of C/N ratio and increasing trend of $\delta^{13}\text{C}$ value during 6.0~9.8 ka, and then becoming current oceanological condition at 6.0 ca. In the last deglaciation, the rapid increasing TOC concentration and $\delta^{13}\text{C}$ value suggest increasing marine biological productivity. In the last glacial maxima, the marine biological productivity was very low and the contribution of terrestrial organic matter increased but the inflow was very little, according to the lowest TOC concentration and $\delta^{13}\text{C}$ value. This study is a part of a development methane hydrate project, Ministry of Economy, Trade and Industry.

Keywords: Paleoenvironmental change, Quaternary, Japan sea, organic carbon, stable isotope

Sediment transportation changes based on spatiotemporal variations of Ni and Cr in the Hida range and off-Joetsu area.

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Submarine sediments in the eastern margin of Japan Sea are presumed to contain materials originated from Japan islands substantially. It is important to clarify transportation and sedimentation processes of the land source materials for understanding formation of methane hydrate component layers. Because Ni and Cr in the submarine sediments in the eastern margin of Japan Sea are considered to be supplied by the Hime river basin (Imai et al., 2010), we can reconstruct the history of sediment transportation from the Hime river basin by the temporal change of Ni and Cr in the sediments. River fed sediments are supposed to deposit on not only submarine canyons but also submarine ridges. This study revealed the temporal changes of Ni and Cr since the last interglacial in three boring cores on submarine ridges. We analyzed MD179-3296 (approximately 45 km distant from Hime river mouth) and 3312 (61 km) cores (approximately depth is 1,000m) close to continental shelf in off-Joetsu area. RC1408 (284km) core drilled in off-Akita-Yamagata area was also examined for a comparison with the MD179 cores. And this study investigated spatial changes of sediment transportation between Hida range and Japan Sea by revealing spatial distribution of Ni and Cr in riverbed sediments of the main river among Hokuriku area and beach sediment of Joetsu area.

The samples were dried and powdered, and then pressed as tablets. Ni and Cr contents were measured by Wavelength Dispersive X-ray Spectrometry (Rigaku Primus II). Age models of 3296 and 3312 cores have been well-established by tephrochronology, radiometric dating, and oxygen isotope ratio of foraminifer (Nakamura et al., 2013; Takizawa et al., 2014, etc). Grain size of river bed sediments of Hime and Oumi river we analyzed by Laser diffraction particle size analyzer (Shimadzu SALD 3000S).

According to spatial distribution of Ni and Cr in geochemical map (Imai et al., 2010), clastic materials from Hime river appear to suspend and be transported by ocean current with gradually sinking within 20~30km from Hime river mouth, and farther than 20~30km, clastic materials appear to flow down by sediment gravity flow. Grain size analysis of river bed sediments of Hime and Oumi river indicates that Ni and Cr are included mainly in coarse grain, and suggests that Ni and Cr are mineral state. Ni and Cr show high concentration near the ultramafic rock, and it is suggested that origin of Ni and Cr is especially restricted in specific parts of Hime and Oumi rivers. Hime and Oumi rivers are steep and have extremely narrow continental shelf around their river mouths, and their source is the northern part of Hida range.

Concentration of Ni and Cr in 3296 and 3312 cores are very similar to each other, and tend to be high (low) during interglacial (glacial) age. However, peaks of Ni and Cr concentration in 3296 core are higher than the other cores. Ni and Cr content rate of RC1408 core is half or one third of 3296 core. Temporal changes of Ni and Cr shows close relation with temperature or rainfall of the Japan Alps estimated from total organic carbon proxy of sediments at the bottom of some lakes (Kumon et al., 2009). Considering topographical steep condition of Hime river, main factor to change concentration of Ni and Cr appear to be changing supply of the serpentine because of changing rainfall influenced by summer monsoon.

This research was a part of METI's project entitled "FY2014 Promoting research and development on methane hydrate".

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Keywords: MD179 marine core, glacial-interglacial cycle, element analysis, Hime river, sediment transportation, summer monsoon

Dissolved gas in pore water at the hydrate-bound field off Sakhalin Island

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We summarize characteristics of dissolved gas in pore water in sub-bottom sediments off Sakhalin Island, where gas hydrates were retrieved. Gas hydrates have been discovered at the northeastern, southeastern and southwestern Sakhalin Island in the cruises of LV31 (2003), LV32 (2003), LV36 (2005), and LV39 (2006) in the framework of hydro-Carbon Hydrate Accumulations in Okhotsk Sea (CHAOS) project, and those of LV47 (2009), LV50 (2010), LV56 (2011), LV59 (2012), LV62 (2013), and LV67 (2014) in the framework of Sakhalin Slope Gas Hydrate (SSGH) project. We retrieved sediment cores (184 cores in total) including gas hydrates (29 cores) in these cruises (R/V Akademik M. A. Lavrentyev).

The dissolved gases in the pore water were extracted according to a headspace gas method. Basically, 10 mL of sediment and 10 mL of saturated aqueous solution of NaCl with a small amount of benzalkonium chloride (preservative) were introduced into a 25 mL vial to create a 5 mL headspace. The headspaces were flushed with nitrogen or helium as an inert gas. The vials were then shaken thoroughly and stored overturned. Molecular and stable isotope compositions of these samples were measured in our laboratory (Kitami Institute of Technology).

The depths of sulfate-methane interface (SMI) distributed mainly 1-3 mbsf, and those of hydrate-bound core were shallower (30 cm to 1 m). The dissolved gases in pore water below the SMI depth were primarily methane (more than approximately 99% of the total hydrate-bound gas), although ethane, propane, CO₂, and hydrogen sulfide were also detected. The molecular and isotopic compositions of dissolved gas in the pore water indicated that the sediment cores including gas hydrates contain ¹³C-rich methane and relatively high concentrations of ¹³C-rich ethane compared to other gas-rich sediment cores. The gas-rich sediment cores without gas hydrates are characterized by high C₁/(C₂+C₃) ratios and ¹³C-depleted methane and ethane. We suggested the idea that small amount of thermogenic gas controlled occurrence of gas hydrate in the subsurface sediment off Sakhalin Island.

Keywords: gas hydrate, pore water, stable isotope, Sea of Okhotsk, Sakhalin Island

Formation of sII gas hydrate during dissociation of sI mixed-gas hydrate composed of methane and ethane

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Double structure gas hydrate, composed of the cubic structure I and II, has been discovered in a same sediment core retrieved from the Kukuy K-2 mud volcano at Lake Baikal (e.g., Kida *et al.*, 2006). The structure II gas hydrate contained 13-15% of ethane, on the contrary, the structure I has only several % of ethane. It has been reported that a structure II gas hydrate appears in appropriate gas composition of methane and ethane (Subramanian *et al.*, 2000a; 2000b). Some models tried to explain how the double structure formed (Hachikubo *et al.*, 2009; Manakov *et al.*, 2013), however, which structure first formed has been still unknown.

Synthetic mixed-gas (methane and ethane) gas hydrates were formed and their dissociation process was investigated by using a calorimeter (Hachikubo *et al.*, 2008). In most cases, two peaks of heat flow appeared and the dissociation process was divided into two parts. This can be understood in the following explanation that (1) the sample contained both crystal structures (I and II), and/or (2) ethane-rich gas hydrate formed simultaneously from dissociated gas and showed the second peak of heat flow. However, Raman spectra of these samples showed that the crystallographic structure of the samples was originally unique in most cases. In this study we tried to check the latter process, namely a secondary formation of ethane-rich gas hydrate.

We made a methane and ethane mixed-gas hydrate (3.62% C₂) in a pressure chamber (volume: 20mL), and stored in liquid nitrogen. A part of the sample was put into a calorimeter (Setaram BT2.15) and dissociated under the temperature gradient of 0.15 K min⁻¹. We observed double peaks in the thermograph, indicating that a new gas hydrate formed from the dissociation gas of the original hydrate, concentrated ethane, and then dissociated. In the next experiment, we put the rest of the sample into the calorimeter, heated in the same condition, and recovered the sample at the end of the first peak of dissociation. The Raman spectra of the sample revealed that a structure II ethane-concentrated gas hydrate formed secondarily in the sample. The composition of ethane was 23.4%, corresponded to the field of structure II (Subramanian *et al.*, 2000a; 2000b). Stable isotopes ($\delta^{13}\text{C}$ and δD) of methane and ethane were also measured, and confirmed the existence of several ‰ difference in ethane δD between the first and second peaks, corresponded to the field data of the double structure observed at the Kukuy K-2 mud volcano (Hachikubo *et al.*, 2009).

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Keywords: gas hydrate, methane, ethane, Lake Baikal