Dissociation properties of natural gas hydrate from the JAPEX/JNOC/GSC Mallik 2L-38 gas hydrate research well by X-ray computerized tomography (CT) experiments

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Abstract: Core samples from the JAPEX/JNOC/GSC Mallik 2L-38 gas hydrate research well contained a variety of forms of gas hydrate within sands and granular sands in the interval from 896 to 926 m. A number of these samples were placed inside pressure vessels charged with nitrogen gas and subsequently transported to Japan for specialized dissociation experiments. X-Ray CT images were acquired, at constant intervals, from a granular sand (collected from 913.76 m) containing pore-space gas hydrate during warming from -35 to 4°C, and subsequently during stepped decreases in cell pressures (0.1 MPa) below assumed threshold stability conditions. Dissociated gas flow and sample temperatures were monitored continuously.

Changes in CT values indicated that gas hydrate dissociated simultaneously, both on the exposed surfaces and within the pore spaces of the sample, in response to pressure changes. This suggested that pressure reductions were effectively transmitted through the sample, most likely because the samples were not fully saturated with gas hydrate. Gas-flow measurements indicated that a larger pressure drawdown caused a higher dissociation rate.

Résumé : Les échantillons carottés prélevés dans le puits de recherche sur les hydrates de gaz JAPEX/JNOC/GSC Mallik 2L-38, dans l'intervalle situé entre 896 et 926 m, renferment diverses formes d'hydrates de gaz contenus dans du sable et du sable grenu. Un certain nombre de ces échantillons ont été placés dans des récipients sous pression remplis d'azote gazeux, puis transportés au Japon pour être soumis à des expériences spécialisées de dissociation. On a acquis, à des intervalles constants, des images tomodensitométriques d'un sable grenu (prélevé à 913,76 m) dont l'espace poral contient des hydrates de gaz au cours du réchauffement allant de -35 à 4 °C et, par la suite, au cours de la baisse échelonnée des pressions cellulaires (0,1 MPa) au-dessous des conditions présumées de seuil de stabilité. L'écoulement du gaz dissocié et la température des échantillons ont été surveillés constamment.

Des modifications dans les valeurs tomodensitométriques indiquent que la dissociation des hydrates de gaz se fait simultanément sur les surfaces exposées et à l'intérieur des espaces poraux de l'échantillon lorsque ce dernier est soumis à des changements de pression. Par conséquent, les réductions de pression seraient en réalité transmises dans les échantillons selon toute vraisemblance parce que ceux-ci ne sont pas entièrement saturés en hydrates de gaz. Les mesures de l'écoulement gazeux montrent qu'une chute plus importante de la pression entraîne une vitesse de dissociation plus élevée.

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INTRODUCTION

This research represents a contribution to the ongoing Japanese methane hydrate research program, with financial support from the Japan National Oil Corporation (JNOC), and ten additional oil, gas, and electric companies in Japan. Gas hydrate is a crystalline substance in which a solid-water lattice accommodates guest molecules of various gases within a clathrate cage. Gas hydrate is widespread in several arctic sedimentary basins associated with deep permafrost, and in deep-water offshore sediments along many coastal margins. Detailed information is lacking about the geologic parameters controlling the occurrence and distribution of natural gas hydrate, especially from a resource viewpoint. Laboratory data for pure-water and methane systems provide general information about methane hydrate stability conditions; however, the formation and dissociation conditions of gas hydrate are known to be influenced by the physicochemical effects of the host strata.

In February and March of 1998, the JAPEX/JNOC/GSC Mallik 2L-38 gas hydrate research well was drilled to the depth of 1150 m at the northeastern edge of the Mackenzie Delta, Northwest Territories, Canada. A drill site adjacent to Imperial Mallik L-38 (an industry exploration well drilled in 1972) was selected for the gas hydrate research well (Bily and Dick, 1974; Collett and Dallimore, 1998; Dallimore and Collett, 1998). Gas-hydrate-bearing core samples, collected from beneath the permafrost interval (from 896 to 953 m) (all depths measured from kelly bushing [8.31 m above sea level]), provided a unique opportunity to study the physical and chemical properties of natural gas hydrate samples.

This paper describes an experiment in which X-ray computerized tomography (CT) scanning techniques were used to observe the dissociation characteristics of a Mallik 2L-38 core sample. Analyses of methane hydrate by the X-ray CT scanner are useful for investigating occurrences of gas hydrate in sediments and determining the relationship of the gas hydrate to the host sediments, without significantly disturbing the core samples. Mallik 2L-38 core samples containing gas hydrate were imaged using X-ray CT scanning techniques before being subjected to further analyses. X-ray CT images were obtained using the Toshiba X-ray CT scanner 'X-force' at the JNOC Technology Research Center. Time-series observations of CT responses during dissociation of gas hydrate within the core sample are presented in this paper. Dissociation forcing was accomplished through a controlled pressure reduction from the pressure-temperature (P-T) equilibrium threshold for gas hydrate stability. Results of this experiment should provide insight into gas production characteristics for natural gas hydrate reservoirs using pressure drawdown techniques.

DISSOCIATION EXPERIMENTS USING THE X-RAY CT SCANNER

The objective of this experiment was to induce controlled gas hydrate dissociation within the test sample by manipulating the drawdown pressure within the test chamber relative to the pressure at which phase equilibrium conditions were previously established. X-ray CT imaging was used to record the changes observed within the core sample during dissociation of gas hydrate.

The core sample utilized for the experiment was a gas-hydrate-bearing granular sand recovered from a depth of 913.76 m. Quantitative well-log derivations (Miyairi et al., 1999) suggested that the sample contained in situ gas hydrate concentrations exceeding 75% pore saturation. When recovered at the surface, the core contained some visible gas hydrate within the pore spaces that occurred as small granules (up to 0.5mm) and coatings on mineral grains (Uchida et al., 1999a). Following retrieval at the drill site, the core sample was placed in a pressure vessel (600 mL capacity) which was then pressurized with nitrogen gas to 6 MPa. The sample was maintained at approximately -70°C for transportation to Japan and subsequently stored at temperatures between -20 and -40°C at the laboratory facilities of the JAPEX Research Center.

The dimensions of the test sample when loaded into the CT scanner were roughly 5 cm by 5 cm by 7 cm, with a bulk volume of approximately 115 cm^3 (Fig. 1). The average porosity of the sample was estimated to be about 30% (Katsube et al., 1999; Uchida et al., 1999b), yielding a total pore volume of about 34.5 cm^3. Given that the specific volume of water in gas hydrate is about 1.26 cm^3/g (Makogon, 1981), the core sample could accommodate a maximum of 27 g of water if the available pore space was fully occupied by gas hydrate. At 100% gas hydrate pore occupancy, the maximum volume of gas that could be incorporated into the gas hydrate within sample pores is about 4800 cm^3 (if 80% of the cages were occupied by guest molecules).

Dissociation apparatus

The experimental apparatus used for the dissociation testing (Fig. 2) was developed by JNOC/JAPEX to be compatible with the X-ray CT scanning equipment. Because X-rays can penetrate aluminum, the core holder was constructed of an aluminum/fibreglass composite (117 mm inner diameter, 135 mm outer diameter, and 360 mm length). It accept samples up to 117 mm in diameter. The core holder is a pressure vessel equipped with a coiled cooling tube jacket. A data acquisition and control system accurately maintained system temperature during the experiments. The core sample was placed on a tray in the core holder, and the system was then pressurized with methane gas. The surface temperature of the sample and the temperature of the outside of the core holder were monitored using thermocouples. A back-pressure regulator located in the gas-discharge line was used to maintain the pressure in the core holder at fixed values. The volume of gas discharged from the core holder was measured using a gas flowmeter.

The X-ray CT analysis was conducted using a Toshiba X-ray CT scanner (model: 'X-force') at the JNOC Technology Research Center. Because the CT scanner was originally designed for medical use on human subjects, the system was modified slightly to facilitate observation of sediment and/or rock samples. The resolution of images is

0.35 square millimetres per pixel and 1 mm in thickness due to the low output voltage of 130 kV. With this configuration, 50 profiles can be obtained within 3 minutes, which facilitates the observation of gas hydrate. However, this resolution is generally too coarse to allow individual components of the core (mineral grains, pore-space hydrate, ice, water, etc.) to be distinguished. Consequently, a mixed or average CT response is obtained at each pixel location, depending on the local composition of the core. Note that CT values ultimately reflect the relative densities of various materials, such that CT values of sediment grains (minerals) usually exceed 1000, while those of common gases diminish to -1000. According to Uchida et al. (1997) and Matsumoto et al. (in press), ODP Leg 164 CT values of methane hydrate range from -100 to -250, whereas those of pure water, pure water ice and dry ice are 0, -65, and 400, respectively. Typical CT values for a variety of materials present in gas hydrate–sediment systems are summarized in Table 1.

Table 1. Typical CT values.

Material	CT value
Free gas	-1000
Methane hydrate	-250 to -100
Water ice	-65
Water	0
Minerals	>1000

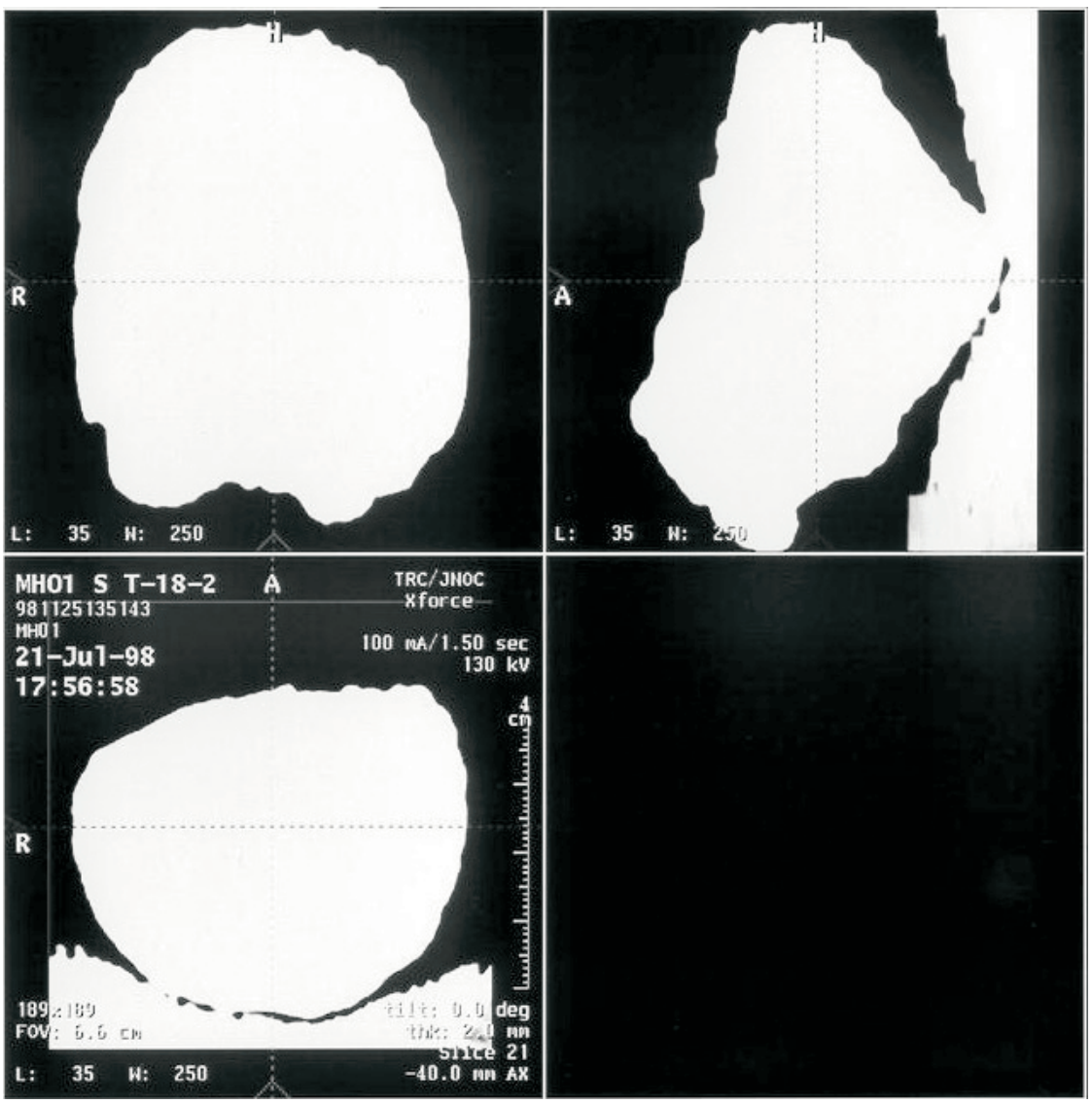


Figure 1. *Profile of the gas-hydrate-bearing sand sample taken along 3 orthogonal axes.*

Experimental procedure

The gas hydrate dissociation experiment was conducted according to the following procedures:

1. The sample was fixed into the core holder, which had been previously chilled to the sample storage temperature of -30°C (Fig. 3). A polycarbonate tray was filled with glass beads (1 mm diameter) in order to support the irregular-shaped core sample. The surfaces of the glass beads were coated with a plastic film to prevent interaction with the sediments and/or dissociated water. Thermocouples were attached to the sample using Teflon® adhesive tape. The sample was placed on a sheet of plastic film and fixed with a nylon sheath to prevent shifting. To minimize sample deformation, care was

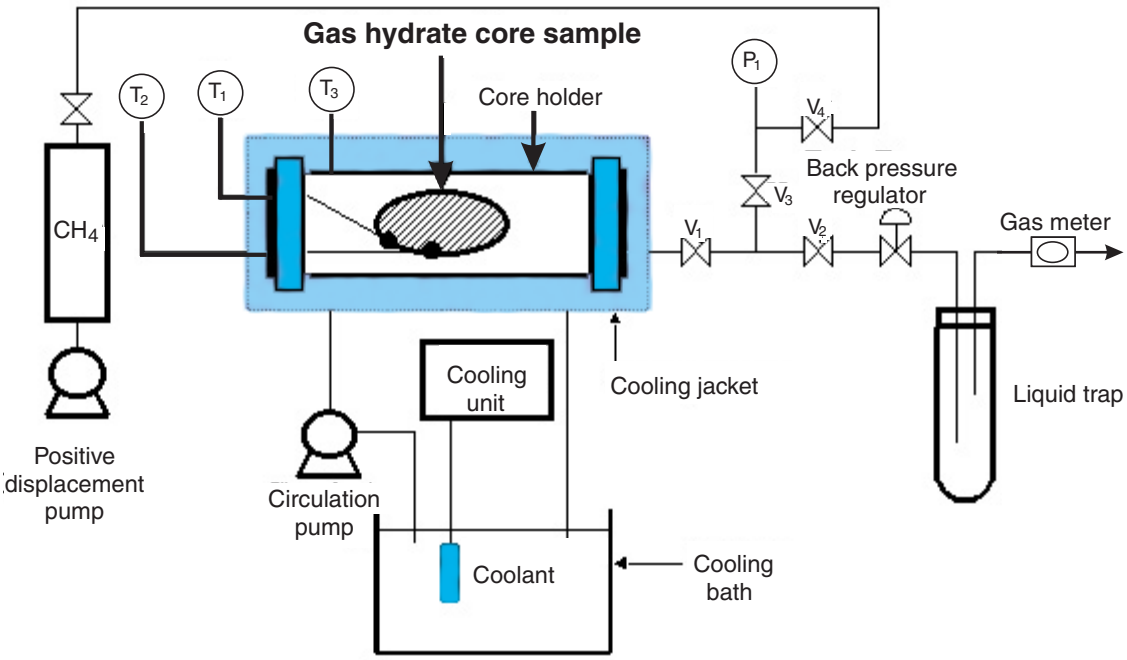


Figure 2. *Schematic drawing of the experimental apparatus. T_1–T_3 are thermocouples, V_1–V_4 are valves, and P_1 is a pressure gauge.*

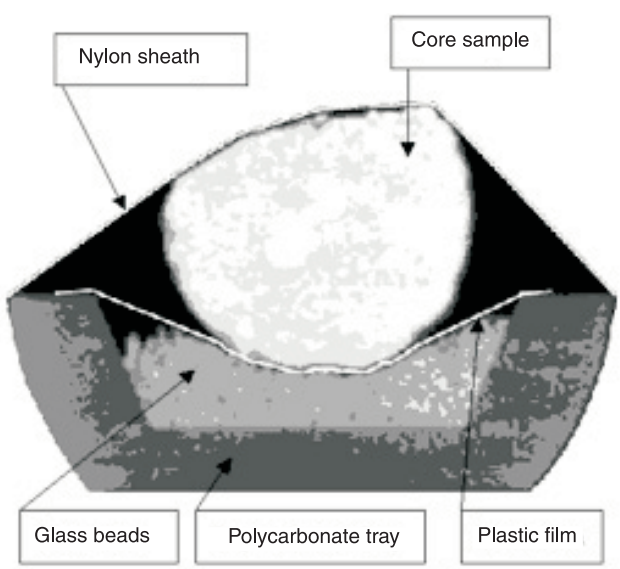


Figure 3.

Fixing method of the sample in the core holder.

taken to ensure that no confining stress was transferred to the core sample. After setting the sample in the core holder, the system was pressurized with methane gas (to approximately 6 MPa) to promote stability of the gas hydrate within the core sample.

2. The core holder was placed on the bed of the X-ray CT scanner and the temperature of the sample was raised gradually in small increments (beginning at about -18°C). During warming of the sample, the outlet valve (V_2, Fig. 2) of the core holder was kept closed and the chamber pressure was continuously monitored. X-ray CT scans were obtained at each temperature step. The sample temperature reached 4°C (the assumed in situ temperature of the host sediments) approximately fifty hours after the start of heating (*see* Fig. 4), after which this temperature was maintained for the duration of the experiment.

3. The outlet valve was opened and the pressure was adjusted to about 0.5 MPa above the hydrate equilibrium pressure anticipated for the specified temperature. After gas flow ceased and the chamber pressure stabilized, the pressure was reduced by an additional 0.05 MPa for several minutes. It was assumed that as the pressure was reduced to a level just below the hydrate equilibrium pressure, dissociation of gas hydrate would begin within the sample. Dissociation subsequently progressed until a constant chamber pressure was re-established, indicating that gas hydrate equilibrium conditions had been achieved (4.3 MPa at 4°C).

4. The core was repressurized (with methane gas) to about 5.7 MPa and maintained at constant temperature (4°C) for a period of 60 hours following the determination of P-T equilibrium conditions. The system pressure was again reduced to 4.3 MPa before beginning the controlled dissociation stage of the experiment.

5. Stepwise pressure drawdowns (from the initial 4.3 MPa stability condition) of 0.1 MPa, 0.2 MPa, 0.3 MPa, 0.5 MPa, and 1.0 MPa were imposed on the sample by bleeding gas from the apparatus, with the intent of inducing dissociation of gas hydrate within the core sample. The pressure drawdown is defined as the difference between the gas hydrate equilibrium pressure and the system pressure controlled by the back-pressure regulator. Additional CT scans were acquired during each pressure-drawdown stage.

RESULTS

During the experiment, the pressure and temperature histories were recorded from the onset of system-temperature increase until the end of the dissociation period (Fig. 4). Figure 5 shows the gas production rates for each dissociation step (the origin on the time axis is the start of the dissociation period). Gas released from the dead volume initially caused a high apparent rate of gas production. Because the dead volume of the core holder was large compared to the sample volume, and given the limited control capacity of the back-pressure regulator, considerable time was required for

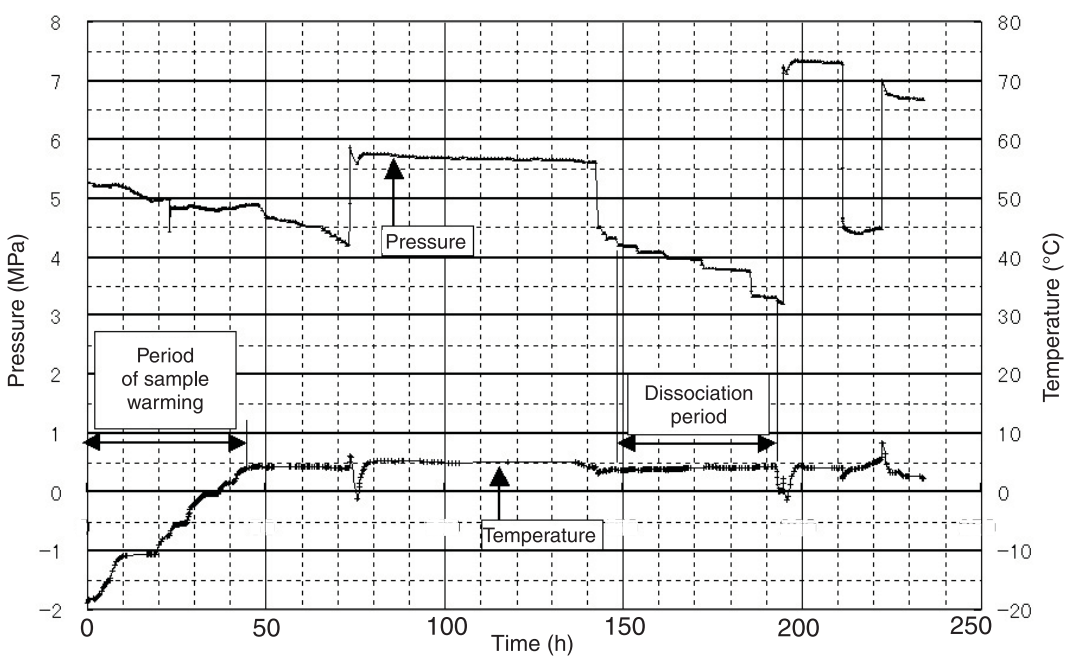


Figure 4. *Pressure-temperature history of the experiment.*

the stabilization of gas-flow rates. Therefore, our estimates of the total volume of methane gas released from the test sample during dissociation of gas hydrate should be considered as rough estimates only.

X-ray CT scans were acquired periodically throughout the experiment. The CT images taken at the initial conditions (-18°C) and after subsequent sample warming to 0°C and 4°C, are shown in Figures 6, 7, and 8, respectively. No significant change was observed during warming from -18 to -5°C. A slight increase in CT values was recognized at around 0°C and a slight decrease in CT values was observed at 4°C (Fig. 9). CT images during dissociation are shown in Figures 10 and 11 and indicate the final state at pressures of 4.0 MPa and 3.3 MPa respectively.

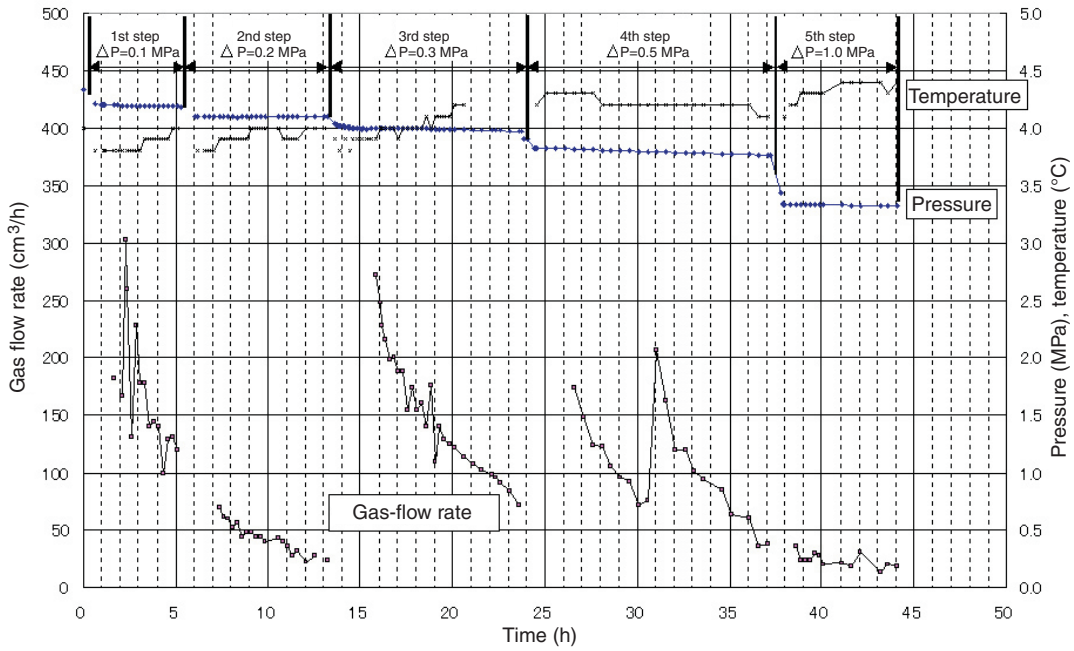


Figure 5. *Pressure, temperature, and gas-flow rate during the dissociation period.*

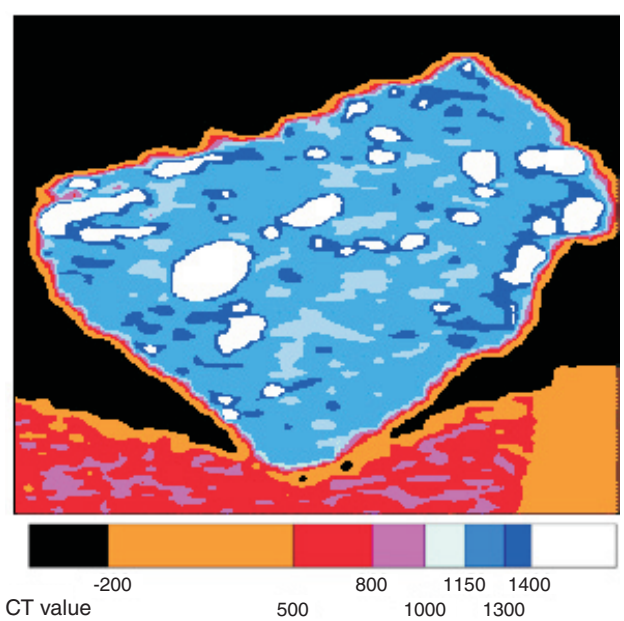


Figure 6. *CT image of sample at T = -18°C (P = 5.2 MPa).*

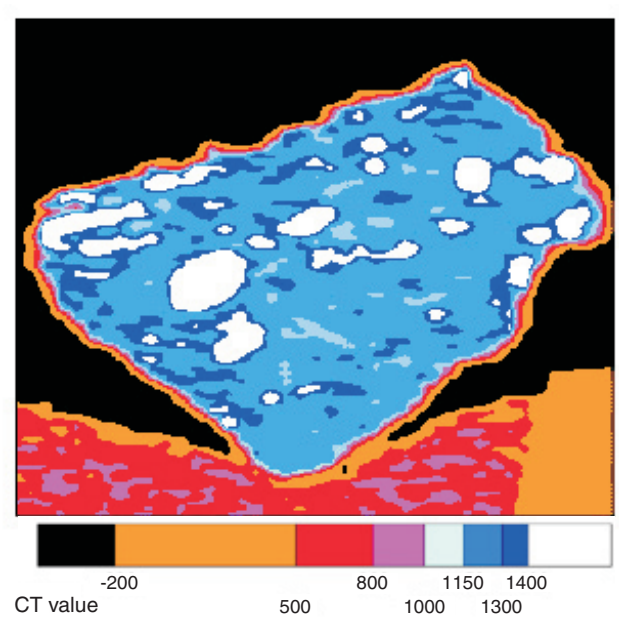


Figure 7. *CT image of sample at T = 0°C (P = 4.8 MPa).*

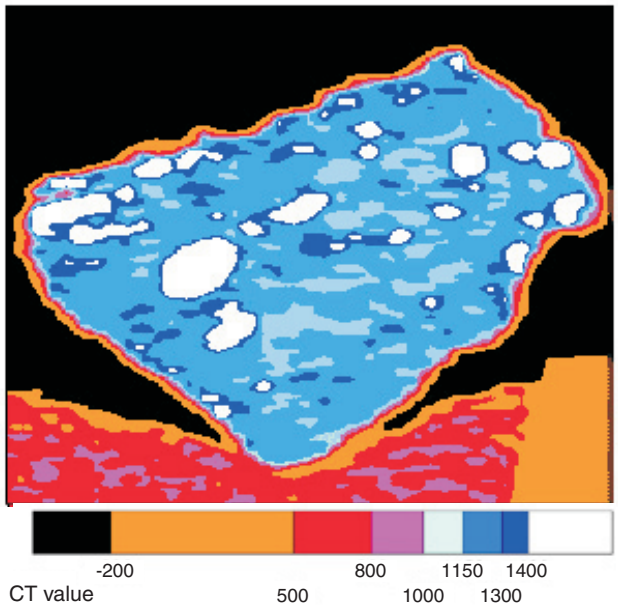
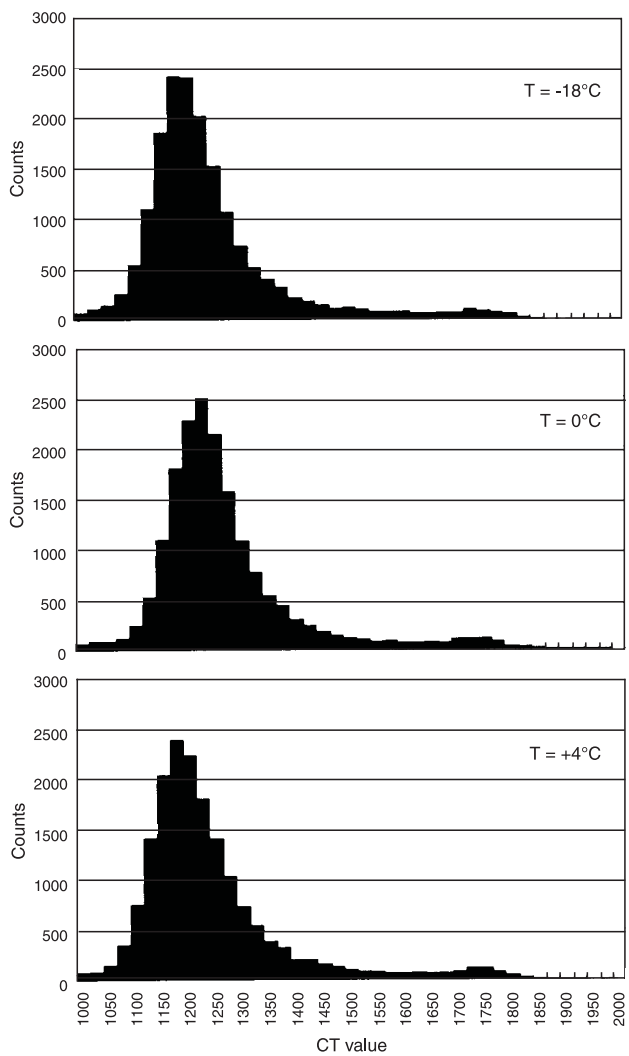


Figure 8. *CT image of sample at T = 4°C (P = 4.9 MPa).*



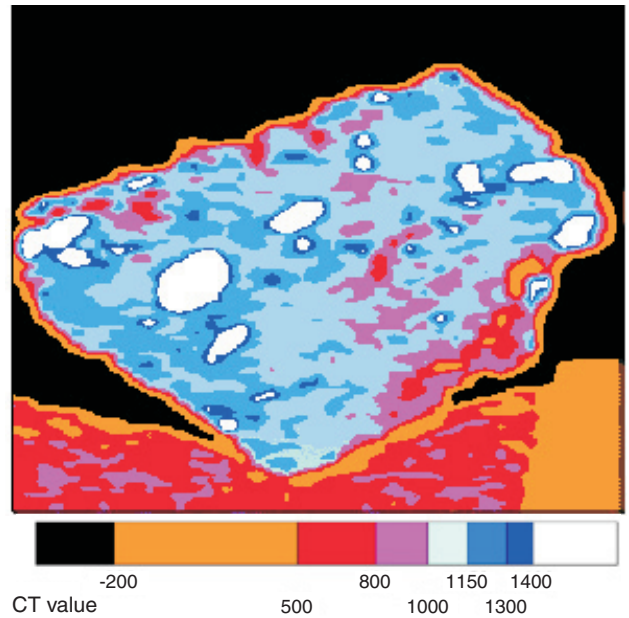


Figure 10. *CT image of sample at P = 4.0 MPa (T = 4°C, ΔP = 0.3 MPa)*

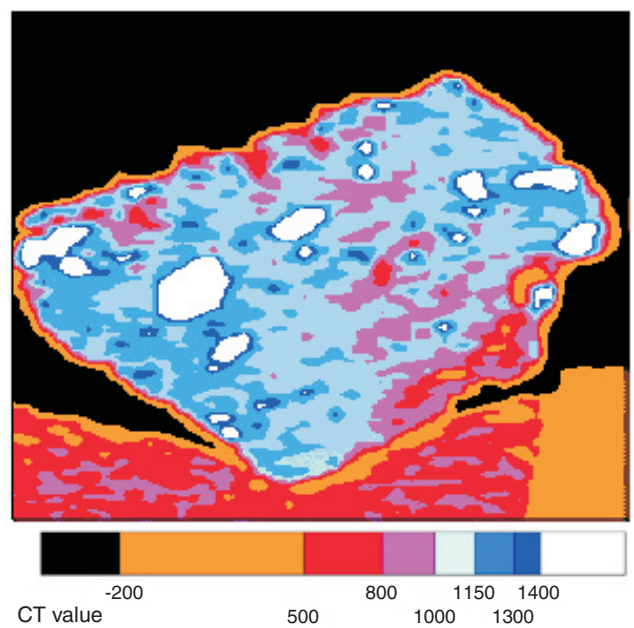


Figure 11. *CT image of sample at P = 3.3 MPa (T = 4°C, ΔP = 1.0 MPa)*

Figure 9.

Frequency change in CT values at -18°C, 0°C, and 4°C. Pressure = 4.8–5.2 MPa.

CT images taken during the gas hydrate dissociation period indicate significant changes in the core during the pressure drawdown of 0.3 MPa. Changes in CT values were less apparent during subsequent pressure drawdowns. Figures 12, 13, and 14 show differential CT values between an image taken prior to the start of dissociation, and three other images (of the same slice) taken after pressure drawdowns of 0.1 MPa, 0.2 MPa, and 0.3 MPa. These figures, therefore, indicate changes in CT values as dissociation progresses within the sample. Warmer colours (oranges and reds) indicate decreased CT values between scans, while cooler colours (blues and violet) indicate increased values. The shift towards more-negative CT values under these stepwise pressure reductions were interpreted as resulting from the presence of additional free gas (CT value near -1000) within sample pore spaces as gas hydrate dissociation progressed (it is assumed that some of the liquid water released as dissociation progressed was pushed out of sample pores).

Figure 12 indicates very little change within the sample under a 0.1 MPa pressure drawdown. Under a 0.2 MPa pressure drawdown, changes in CT values are more apparent within the interior of the sample (Fig.13). The most extensive changes in CT values within the sample interior were observed under a pressure drawdown of 0.3 MPa (Fig. 14), which were also indicated by the frequency change observed between the scans obtained at chamber pressures of 4.3 MPa and 4.0 MPa (Fig. 15). Differential CT images could not be obtained at drawdowns of 0.5 MPa and 1.0 MPa because the sample moved slightly during dissociation under the 0.3 MPa pressure drawdown. The CT-scan results suggest that gas hydrate dissociation in this porous, sandy, core sample occurred both at the sample surface and also within the interior of the sample.

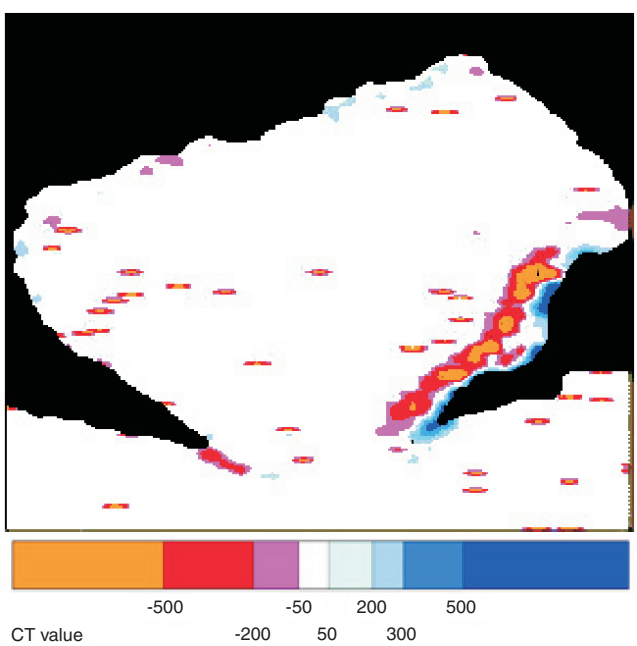


Figure 12. *Differential CT values of sample at ΔP = 0.1 MPa (T = 4°C, P = 4.2 MPa)*

The rates of gas production at each pressure drawdown are plotted in Figure 16. Because the duration of the first dissociation step (the dissociation period at pressure drawdown of 0.1 MPa) was not enough to stabilize the flow rate, these data were included in the second step. At the fourth step (pressure drawdown of 0.5 MPa) the gas flow rate was divided into

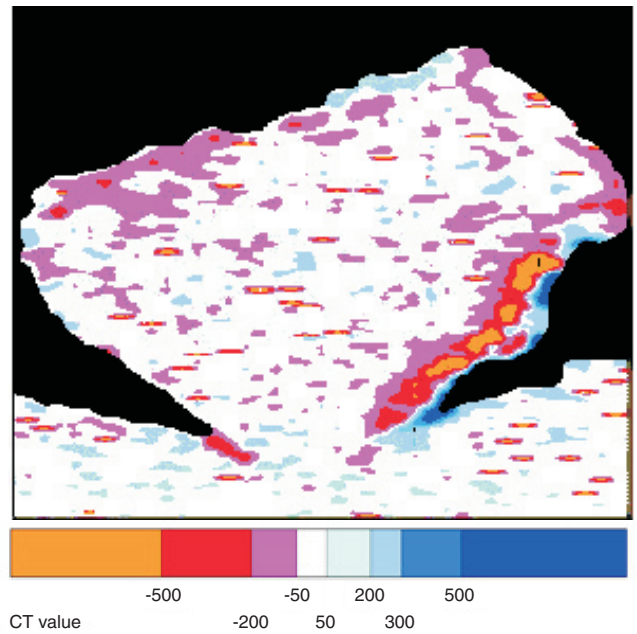


Figure 13. *Differential CT values of sample at ΔP = 0.2 MPa (T = 4°C, P = 4.1 MPa)*

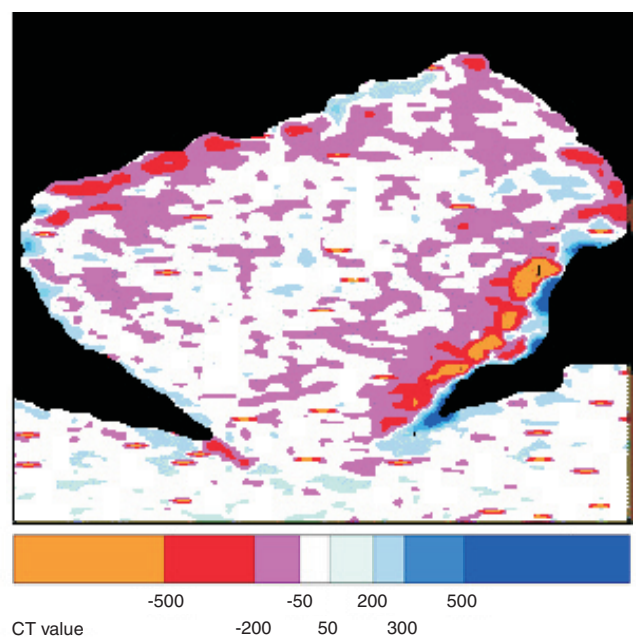


Figure 14. *Differential CT values of sample at ΔP = 0.3 MPa (T = 4°C, P = 4.0 MPa)*

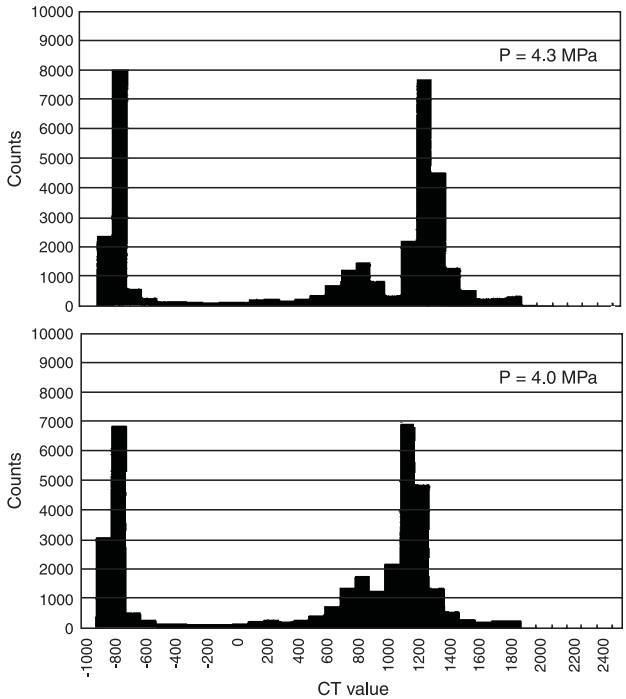


Figure 15. Frequency change in CT values at 4.3 and 4.0 MPa.

two stages, due to the necessity for adjustment of the back-pressure regulator in the middle of this step. Because the back-pressure regulator had insufficient flow capacity, excessive time was required to discharge the gas in the dead volume pressure chamber, and a stabilized gas-flow rate was not obtained. Comparison of gas-flow rates from the second and third step indicate very different gas-production rates (40 cm^3/hr and 80 cm^3/hr respectively, after 10 hours). Gas-flow rates in subsequent drawdowns of 0.5 MPa and 1.0 MPa were generally much reduced, suggesting that most of the gas hydrate in the core sample had dissociated during the previous pressure drawdowns.

DISCUSSION

The nature of gas hydrate dissociation at atmospheric pressure conditions and temperatures between 3 and -5°C was recorded on film and video immediately after core recovery at the drill site. Within the coarse-grained (sandy to pebbly) sediments characteristic of the Mallik 2L-38 well, gas hydrate apparently dissociated relatively slowly, with continuous bubbling of methane gas observed on the exposed surfaces of recovered core sections. Though generally less common, in some instances visible gas hydrate grains (2–5 mm) dissociated vigorously, with the rapid release of gas causing popping sounds and propelling small grains considerable distances. Even at laboratory temperatures (near 20°C) the gas hydrate was relatively stable and rarely

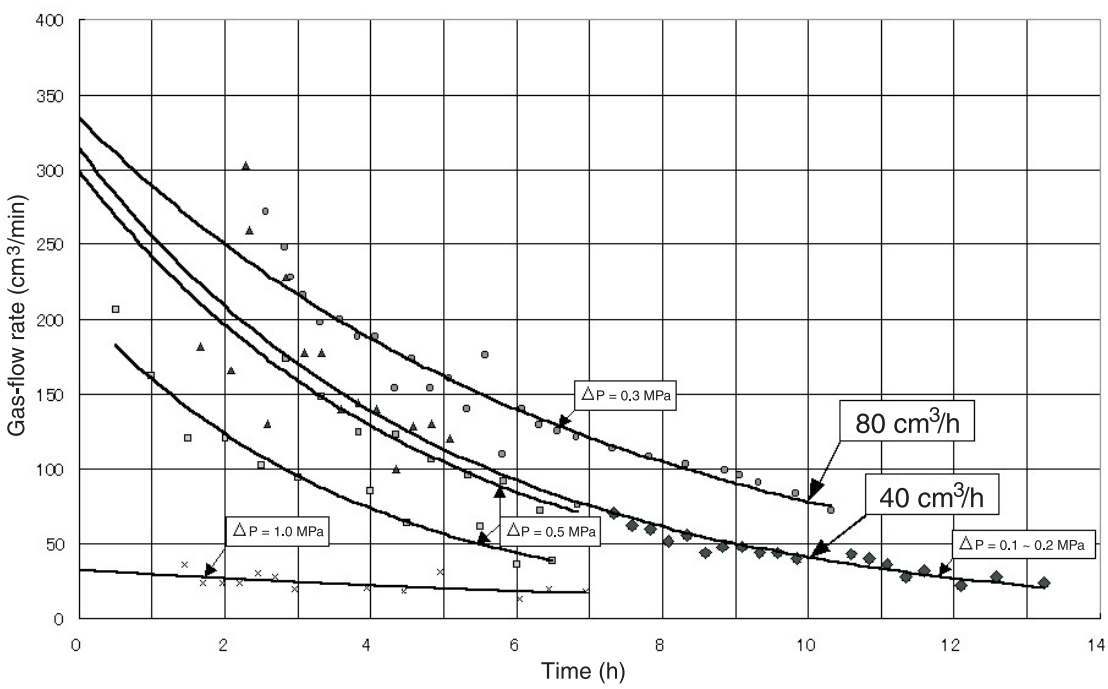


Figure 16. Gas-flow rate vs. time during pressure drawdown and gas hydrate dissociation .

generated sufficient gas to support combustion. We believe this behaviour may be partly due to the self-preservation phenomena (Ershov and Yakushev, 1992), whereby gas hydrate dissociation is impeded by the formation of an ice coating (facilitated by the endothermic dissociation reaction) on gas hydrate surfaces. The role of this ice phase, either within the sediment pores or as a coating on the surface of the gas hydrate itself, may be critical in some gas hydrate dissociation situations.

We have considered the effects of increasing temperature on gas hydrate dissociation. The core sample was initially stored in a freezer at temperatures below -30°C. No changes were observed in the sample as during warming from -18 to -5°C, with CT values remaining almost constant during this period. However, upon further warming, CT values were found to increase slightly around 0°C. The CT values for freshwater ice are typically around -65 while fresh water is around 0 (Table 1). We attribute this slight increase in CT values to be a result of ice melting to water within the sample. Free water is believed to have initially coexisted with gas hydrate within sediment pores, and this water was converted to ice during sample storage in the freezer. A slight decrease in CT values was observed at a temperature of 4°C (at about 5.7 MPa), and this may have been due to the formation of additional gas hydrate within the sample between hour 75 and hour 140 (Fig. 4). This conclusion is supported by the slight decrease in pressure observed during this period. The formation of additional gas hydrate should result in a lower net CT value, given that the CT value for pure gas hydrate (-100 to -250, Table 1) is lower than that of the free water (CT value around 0) from which the gas hydrate formed.

The effects of pressure reduction on gas hydrate dissociation was investigated through a series of step-wise pressure reductions. The gas hydrate dissociation process was observed at each pressure drawdown from 0.1 MPa to 1.0 MPa. After the fourth step (0.5 MPa pressure drawdown), gas production decreased and little change was observed in the CT images. The system pressure also showed a tendency to decrease after the fourth step. These results indicate that almost all the gas hydrate in the sample was dissociated before the end of the third step (0.3 MPa pressure drawdown), and that only a small amount of gas hydrate remained in the sample after the fourth step.

The amount of gas dissociated up to the end of the third step was calculated to be about 3500 cm^3, based on the flow rates during the period in which headspace effects were considered to be no longer a factor (i.e. after about 10 hours). The theoretical maximum volume of gas which could be incorporated into gas hydrate within the sample is about 4800 cm^3 (at 80% cage occupancy), suggesting that approximately 73% of pore space was occupied by gas hydrate prior to initiation of dissociation. Note that these results cannot be related to in situ gas hydrate concentrations due to the storage history of the samples, and the fact that the test core was maintained at P-T conditions favouring methane hydrate formation for a period of some 70 hours prior to the initiation of gas hydrate dissociation.

The following interpretations were made based on the observations:

1. Sediment pores were not completely filled with gas hydrate and therefore some pressure conduits existed in the sample. At temperatures above 0°C, free water can co-exist along with gas hydrate in sediment pores. Free water can serve as a pressure conduit to transmit pressure changes into the interior of the sample, thereby initiating dissociation of gas hydrate under isothermal conditions. Given that the sample was chilled to between -70 and -35°C for transportation and storage, any free water existing in sediment pores at the time of recovery would have been frozen prior to the start of the experiment. The slight increase in CT values as the sample was warmed above 0°C is consistent with this interpretation.
2. The sediment fabric can be deformed by repeated fluctuations in pressure, particularly if the gas hydrate is serving as the primary bonding agent between sediment grains. Note that during the experiment the sample was exposed to gas pressure only, without any simulated overburden (confining) pressure. Therefore, flow paths could more readily be kept open once the bonding agents were disrupted.

We initially hypothesized that dissociation of gas hydrate should begin at the surface of the sample and advance inward. CT scans suggest, however, that dissociation actually occurred simultaneously within the interior and on the surface of the sample. It cannot be concluded, however, that this phenomenon is characteristic of gas hydrate dissociation within sediments. Field observations suggest that a considerable portion of the gas hydrate originally present in recovered cores had dissociated during the recovery operations, significantly changing the characteristics of the core sample relative to its in situ condition. Transportation and storage conditions may also have affected physical properties of the samples. Additional investigations are needed to clarify the affects of different transportation and storage methods on the properties of gas-hydrate-bearing sediments. The core-holder, sample-fixing method should be improved because some of the differential CT images could not be obtained between runs due to slight movement of the sample. Gas production from gas hydrate dissociation under a 0.3 MPa pressure drawdown may be double the rate observed for 0.2 MPa drawdown under pseudo-stabilized conditions. This suggests that higher drawdown pressures may cause higher dissociation rates for gas hydrate in sediments. Further modifications of the experimental apparatus are needed to enable more precise analysis, but these data are valuable as a comparison with future simulation studies.

SUMMARY AND CONCLUSION

The dissociation of gas hydrate in a sand-dominated core sample from the Mallik 2L-38 well site was investigated using X-ray CT scanning techniques. During the experiment, time-series CT images of the interior of a gas-hydrate-bearing sediment sample were successfully acquired. The CT images obtained during sample warming indicate that an increase in

CT values at 0°C is related to the melting of pore ice formed from free water that initially co-existed with gas hydrate in the sediment pores prior to freezing of the samples for transport and storage. A decrease in CT values at about 4°C may be indicative of additional gas hydrate formation prior to initiation of the controlled-dissociation stage, but this is still under investigation.

Changes in CT values during forced dissociation indicate that gas hydrate dissociation was initiated nearly simultaneously within the interior of the sample and at the sample surface. It is believed that sample pores were not completely filled with gas hydrate, and therefore may act as conduits for the transmission of pressure reduction. Transportation and storage conditions may have also affected the nature of the sample.

The rate of gas production during the dissociation period can be calculated with limited accuracy only due to a number of experimental uncertainties mentioned earlier. Gas-flow measurements suggest, however, that a larger pressure drawdown causes a higher rate of dissociation of gas hydrate within the sediment pore volume.

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