論 説
Original Article

Characterization of permeability and thermal conductivity of methane hydrate sediments using microfocous X-ray computed tomography technique

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(Received February 9, 2009 ; accepted July 2, 2009)

Abstract : Permeability and thermal conductivity of natural gas hydrate sediments were characterized using a microfocus X-ray computed tomography (CT) technique. The free-gas spaces, sand particles, and hydrates or ices were identified from the obtained three-dimensional X-ray CT images, from which continuous pore networks were identified. The absolute permeability of the sediment correlated well increased with the ratio of parallel to vertical directions of continuous pore numbers. This fact indicates that the pore network in sediment can be useful for assessment of permeability. In order to characterize the thermal conductivity of methane hydrate sediments, a homogenization method was applied to microfocus X-ray CT images. The thermal conductivity calculated from three dimensional X-ray CT images was good agreement to those measured by the hot disk method, when the thermal conductivity of sand particles is 0.5 W/mK. These results demonstrated that the microfocus X-ray CT imaging can be effective for studying material flow and thermo-mechanical properties of natural gas hydrate sediments.

Keywords : permeability, thermal conductivity, X-ray computed tomography, homogenization method, natural gas hydrate sediment

1. Introduction

Gas hydrates have been recognized as an unconventional natural gas resource because huge amount of natural gasses are stored in crystalline hydrates in deep-sea sediments and in permafrost (Max, 2000). Several research wells have been drilled to discover natural gas hydrate in oceanic and permafrost sediments, and the distribution of the gas hydrates was confirmed. The production of natural gas as a new energy resource is currently developed using such methods as depressurization, thermal stimulation, and injection of hydrate inhibitors (Moridis, *et al.*, 2004). It is important to understand the physical properties of sediment in investigations of structural properties, such as permeability, hydrate saturation, and sediment porosity. Since the porosity is particularly important for material flow. Many have been reported on the relationship between porosity and permeability (Arns, *et al.*, 2005 ; Bernabe, *et al.*, 2003 ; Noiriel, *et al.*, 2004 ; Pape, *et al.*, 2000 ; Pape, *et al.*, 2005 ; Quispe, *et al.*, 2005 ; Singh, *et al.*, 2000). Gas and water can flow in serial pores in sediment. Thus the investigation of the pore network is strongly necessary to understand the gas-water flow in the sediment. Studies of the pore network are essential not only to petroleum engineering

but also to chemical engineering in filtration processes (Lin and Miller, 2000 ; Lin and Miller, 2004). A thermal conductivity of hydrate sediments is also important for the production of natural gas from the hydrate reservoir regarding to heat release at hydrate dissociation. In fact, many reports have been published on the theoretical and experimental investigations of thermal conductivity of hydrate sediments (Kumar, *et al.*, 2004 ; Huang and Fan, 2005 ; Waite, *et al.*, 2002 ; Gupta, *et al.*, 2006 ; Revil, 2000).

Microfocus X-ray CT is useful for investigating the small-scale features of objects since it offers greater spatial resolution. In previous studies, microfocus X-ray CT technique has been applied to nondestructively structural investigations of hydrate sediments. The porosity and hydrate saturation of hydrate sediments were directly determined from the X-ray CT images within an accuracy of 3–8% without destroying the samples (Jin, *et al.*, 2004 ; Jin, *et al.*, 2006). Thus the analysis of continuous pore network regarding to material flow can be achieved from a three-dimensional (3-D) image. Furthermore, Ichikawa *et al.* (2001) have been investigated a mechanical properties of granitic rock by a homogenization method (HM) used to the X-ray CT images. The HM is the one of the three dimensional finite element methods (FEM), which can analyze elastic and thermal properties of media (Sanchez-Palencia and Zaoui, 1987). The formulas of the Young's modulus, Poisson's ratio, thermal expansion coefficient, and thermal conductivity

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for the HM have been conducted by Takano and Zako (2000). Shabana and Noda (2008) have successfully performed a numerical evaluation of the thermo-mechanical properties of a functionally graded material (FGM). The X-ray CT can apply to directly and nondestructively obtain the divided area for MH calculations. In this paper, we present the relationship between the continuous pore, which allows gas and water flow, and the absolute permeability of natural methane gas hydrate sediment based on 3-D X-ray CT image data. The pore network provides the predominant factor of the material-flow process in natural sediments. Also the observed 3-D X-ray CT images on artificial and natural gas hydrate sediments are applied to the HM to evaluate the thermal conductivity. Preliminary evaluation results are compared to the thermal conductivity measured by a hot disk method.

2. Experimental

2.1 Hydrate sediment samples

Natural-gas hydrate sediments were sampled from the wells bored by the Japanese government's Ministry of Economy, Trade and Industry (METI) in 2004. These wells were drilled at water depths between 700 and 2,000 m in the north eastern Nankai Trough ca. 50 km off Tokai, central Japan. The detailed treatment of natural gas hydrate sediment core samples for the CT imaging was presented in elsewhere (Jin, *et al.*, 2007). A few natural hydrate sediments have been imaged to permeability and thermal conductivity. Five specimens of natural gas hydrate sediments were measured by microfocus X-ray CT to analyze the relationship between continuous pore network and permeability, which are labeled the samples A, B, C, D, and E. For characterization of thermal conductivity, two natural gas hydrate cores labeled samples F and G, were examined. Also two types of artificial methane hydrate sediments were prepared. One was prepared by compressing of mixture of sand particles and methane hydrate particles (labeled sample H), other was fabricated by methane gas flowing into sand particles saturated by water at 243 K and 10 MPa (labeled sample I). The details of artificial hydrate sediments were described in previous reports (Yamamoto, *et al.*, 2004 : Chuvilin, *et al.*, 2003). A part of core samples cut for structural observation by the X-ray CT. The remaining other parts of the sediment samples were used for the thermal conductivity measurement.

2.2 Microfocus X-ray CT imaging

The structural images of hydrate sediments were obtained using a microfocus X-ray CT apparatus (SMX-225CTS-SV, Shimazu Co Ltd. Japan). A schematic of the microfocus X-ray CT apparatus and the detailed conditions of imaging were previously described by Jin *et al.* (2004). Samples were set in a cylindrical insulated holder mounted on a precision rotation table. The samples of cylindrical 5-mm-diameter were kept below the decomposition temperature of hydrates (~ 173 K) by blowing cold, dry nitrogen gas above the samples. CT raw data measurements were performed at the source voltage of 70kV and in a current range from 40 to 50 μ A. The acquired structural 3-D CT images of sediments were analyzed using

image-processing software (TRI/3D-VOL, Ratoc System Engineering Co. LTD., Japan). The 3D images were decomposed into sand particles, ice, hydrate, and free-gas spaces by the way of analysis of the gray-scale histogram as previously reported (Jin, *et al.*, 2006). Porosity and hydrate saturation were calculated from the volume fraction ratio of each decomposed elements by X-ray CT imaging. The details of their estimation were described in previous reports (Jin, *et al.*, 2004 : Jin, *et al.*, 2006).

2.3 Permeability and thermal conductivity measurements

The absolute permeability measurement of the natural gas hydrate sedimentary samples was performed using a hydrostatic tri-axial pressurized system. The frozen, shaped sediment was set in a cylindrical rubber sleeve and pressurized by methane gas. The inner-core water pressure was kept at 14.0 MPa, and the confining pressure was 15.2 MPa. The water-flow rate was 0.001 to 1.0 cm^3/min and the temperature was at 283 K. The inner-core pressure decreased to the lower equilibrium pressure of methane-hydrate formation with a decreasing confining pressure. The absolute permeability of the sediments was measured based after the decomposition of methane hydrate.

Thermal conductivity of methane hydrate sediments was measured by a hot disk probe system (TPA-501, Kyoto Electronics Manufacturing Co. LTD., Japan). A sensor element shaped of a 10-mm-thick Nickel-metal double spiral sandwiched by two polymethyl methacrylate made a contact with sample. The detail of the measurement of thermal conductivity using this technique was explained by Yamamoto *et al.* (2004).

2.4 Thermal conductivity estimation by homogenization method

In the HM, the original medium is divided into areas consisting of heterogeneous microstructures. In one divided area, it is assumed to consist of homogeneous and periodic cubic unit cells. The schematics definition of divided and unit cell for HM is presented in Fig. 1. Here, for the macroscale x and microscale y as shown in Fig. 1, the scale ratio is defined by

$$\bar{y} = \bar{x}/\epsilon. \quad (1)$$

The heat conduction problem is described and solved using the equations proposed by Takano and Zako (2000). Thermal conductivity calculation procedure by Takano and Zako (2000) is briefly described below. Assuming the neglect of inflow of heat on boundary from outer heat source, the heat conduction

Fig. 1 Schematic definition of the macroscale and the microscale in HM.

problem can be described by :

$$K_{ij} = \frac{\partial T}{\partial x_j} \frac{\partial \delta T}{\partial x_j} d\Omega = \int_{\Omega} f \delta T d\Omega, \quad \forall \delta T \quad (2)$$

where K_{ij} is the thermal conductivity, f is the internal heat source, δT is the temperature gradient for each coordination. The temperature field $T(\vec{x}, \vec{y})$ is defined by relationship as follows :

$$T(\vec{x}, \vec{y}) = T^0(\vec{x}) + \varepsilon T^1(\vec{x}, \vec{y}), \quad (3)$$

where T^0 and T^1 are the macroscopic and microscopic temperatures, respectively. The following averaging principle for certain periodic function Ψ can be adopted in the relationship between macroscale and microscale :

$$\lim_{\varepsilon \rightarrow 0} \int_{\Omega} \Psi \left(\frac{\vec{x}}{\varepsilon} \right) d\Omega = \int_{\Omega} \frac{1}{|Y|} \int_Y \Psi(\vec{y}) dY d\Omega, \quad (4)$$

where $|Y|$ is the volume of the unit cell. Thus, from Eqs. (2) – (4), the heat conduction equations in the macroscale and microscale are derived respectively, as

$$\int_Y K_{ip} \frac{\partial \phi^j}{\partial y_p} \frac{\partial \delta T^1}{\partial y_j} dY = \int_Y K_{ij} \frac{\partial \delta T^1}{\partial y_j} dY \quad \forall \delta T^0, \quad (5)$$

$$\int_{\Omega} K_{ip}^H \frac{\partial T^0}{\partial x_j} \frac{\partial \delta T^0}{\partial x_i} d\Omega = \int_{\Omega} f^H \delta T^0 d\Omega. \quad (6)$$

By solving the microscopic Eq.(5) using the periodic boundary condition, the characteristic parameters such as K_{ip} and K_{ij} . Thus, in Eq. (6), we can obtain the homogenized thermal conductivity tensor and internal heat source as follows :

$$K_{ip}^H = \frac{1}{|Y|} \int_Y \left(K_{ij} - K_{ip} \frac{\partial \phi^j}{\partial y_p} \right) dY, \quad (7)$$

$$f^H = \frac{1}{|Y|} \int_Y f dY. \quad (8)$$

The commercial software for the HM (VOXELCON, Fujitsu Co. LTD., Japan) is used to calculate the thermal conductivity from Eqs. (5) to (7), using the decomposed structural 3-D CT images.

3. Results and Discussion

3.1 Permeability of natural gas hydrate sediments

Fig. 2 depicts a 3-D X-ray CT image of sample A with the fields of view of 3.0 mm for the XY-plane and 2.0 mm for Z-plane, respectively. Table 1 lists the properties and the

Fig. 2 3-D X-ray CT image of sample A. The XY plane of FoV was 3.0 mm and the Z plane was 2.0 mm. The arrow indicates the permeability measurement direction. (Reprinted from Jin *et al.*, (2007) in the Japanese Journal of Applied Physics with permission of The Japan Society of Applied Physics.)

absolute permeability of the samples. The porosity data from the 3-D CT image were in good agreement with those estimated by the mercury porosimeter. Fig. 3(a) represents a cubic (1.2^3 mm^3) part cut from the white square region in Fig.2. Fig.3 (b) represents the continuous pore network (channel) in the cubic part as shown in Fig. 3(a), which allows gas and water flow. The horizontal and vertical channels corresponded to the continuous pore networks distributed to parallel and normal directions to the materials flow during the measurement of the absolute permeability. The horizontal channel of samples was obtained by the analysis of the continuous pore spaces from the top to bottom faces in the cube as shown in Fig. 3(c). The resulting horizontal-channel data consisted of numbers at the top and bottom faces in the cube is listed in Table 1, in addition to the obtained permeability data. We assessed the effect of channel number on the permeability. Since the area of X-Y plane is $1.2^2 = 1.44 \text{ mm}^2$, the horizontal-channel densities were calculated from the dividing the average number of horizontal channels listed in table 1 by the area of X-Y plane of cubic CT images. The absolute permeabilities of the sediments are plotted against the horizontal-channel density in Fig. 4. The horizontal-channel density was the average of horizontal-channel number at the top and bottom faces in flow direction (the arrow as indicated in Fig. 3(a)), which is normalized by the face area of a treated cubic 3-D image. The absolute permeabilities increased with increase in the channel density.

It was possible that the gas-water flow in the sediment was

Table 1 Structural properties of natural methane hydrate sediments.

Sample	Porosity (%)		Absolute Permeability (mD)	Hydraulic conductivity (m/s)	Channel number	
	by 3-D image	by Mercury porosimeter			Top	Bottom
A	43.8	44.2	0.99	7.31×10^{-7}	295	392
B	46.8	40.0	1.12	8.27×10^{-7}	431	558
C	44.4	38.2	14.59	1.08×10^{-5}	434	451
D	43.8	47.5	20.79	1.54×10^{-5}	588	846
E	46.1	44.4	11.93	8.81×10^{-6}	380	581

Fig. 3 Cubic 3-D image cut from white square region of Fig.2. (a) 3-D image for analyzing the continuous pore. (b) The continuous pore image in the cubic 3-D image. (c) The horizontal continuous pore forms from the top to bottom face. (Reprinted from Jin *et al.*, (2007) in the Japanese Journal of Applied Physics with permission of The Japan Society of Applied Physics.)

Fig. 4 Correlation between absolute permeability and horizontal-channel density. (Reprinted from Jin *et al.*, (2007) in the Japanese Journal of Applied Physics with permission of The Japan Society of Applied Physics.)

Fig. 5 Correlation between absolute permeability and the ratio of horizontal- to vertical-channel numbers. (Reprinted from Jin *et al.*, (2007) in the Japanese Journal of Applied Physics with permission of The Japan Society of Applied Physics.)

in both horizontal and vertical directions, because the pore in sediments can extend in any direction. The vertical-channel data were calculated by analyzing the continuous pore spaces between the side faces in a vertical direction. Namely, the horizontal channel density was calculated from the area of X-Y plane and the channel numbers between X-Y planes. The vertical channel density was obtained from the average channel densities between the X-Z planes and Y-Z planes. On the one hand, the vertical-channel density in samples C, D, and E were all close to the horizontal-channel density and the absolute permeabilities increased with increases in the vertical-channel density. On the other hand, samples A and B had approximately twice the horizontal-channel density. Fig. 5 plots the absolute permeabilities of sediments against the ratio of horizontal- to vertical-channel density. Samples with higher absolute permeability shifted to a higher ratio of horizontal-channel density, while samples with lower absolute permeability shifted to a lower ratio. The density ratio of horizontal to vertical directions indicates somewhat more flow probability in the horizontal direction. The absolute permeability increases with a higher ratio of horizontal channels to vertical channels. The structure of the channel network plays the most dominant

role in material flow in natural-gas hydrate sediment. The relationship between the permeability and grain-size distribution were previously reported by Jin *et al.* (2007). Thus the channel network strongly depends on the pore-size or grain-size distribution of sedimentary samples.

3.2 Thermal conductivity of gas hydrate sediments

In Fig. 2 and Fig. 3(a), compositional elements in sediment were composed into sand particles (gray), hydrate/ice (dark gray), and free-gas spaces (black), respectively. The histogram of gray-scale values of sample F is shown in Fig. 6(a). This histogram only two peaks because the resolution of microfocus X-ray CT is not high enough for four-component analysis. Here, we can suppose that this histogram is composed of four Gaussian peaks caused by pore space ($\sim 0 \text{ g/cm}^3$), CH_4 hydrate ($\sim 0.91 \text{ g/cm}^3$), ice ($\sim 0.93 \text{ g/cm}^3$), and sand ($\sim 2.4 \text{ g/cm}^3$). Sato *et al.* (2005) pointed out that the gray-scale value is proportional to the density of material in hydrate sediments on X-ray CT imaging, although the difference in the gray-scale is caused by the difference in the absorption coefficient of X-ray, which depends on the X-ray source voltage, the beam hardening effect, the relationship between sizes of component

Fig. 6 Histogram of gray-scale of sample F. (a) Original histogram. (b) Processed histogram.

and unit cell. Thus Gaussian functions were arranged to gray-scale positions corresponding to their densities, as described in previous report (Jin, *et al.*, 2006). The ratios of the integrated intensities of the Gaussian peaks corresponding to a pore, CH_4 hydrate, ice and sand as shown in Fig. 6(b) is used to determine the volume fractions of each component. The unit cell to solve the microscopic equation (5) consists of pore, hydrate, ice, and sand regions with these volume fractions. The volume fractions of sand, methane hydrate, ice and pore of sample F are obtained to be 0.47, 0.05, 0.28, and 0.20, respectively. When the thermal conductivity values of sand, methane hydrate, ice, and pore are 0.5, 0.5, 3.0, and 0.024 W/mK, respectively, the averaged homogenized thermal conductivity value $K_{11}^H = 1.570$ W/mK was obtained by solving macroscopic equation

(7). On the other hands, the simple average value of thermal conductivity calculated from these volume fractions and each thermal conductivity values is 1.105 W/mK. Thus the homogenized value agreed well to the thermal conductivity value (1.766 W/mK) measured by the hot disk method.

Similar 3-D X-ray CT imaging and HM calculations were carried out in all testing sedimentary samples. Table 2 listed the thermal conductivities of sedimentary hydrate samples calculated from X-ray CT images (observed at 173 K) and measured by the hot disk method at 143 K. In this table, the porosities and hydrate saturations of sedimentary hydrate samples determined from mass and gas volume measurements and X-ray CT gray-scale analysis. Thermal conductivity values obtained from X-ray CT imaging agree with those of obtained from direct measurements in an accuracy of 20%.

Toyoura standard sand is mainly composed of quarts, of which the thermal conductivity of single crystal of quarts is ~ 6.2 W/mK at 273 K (Yamamoto, *et al.*, 2004). On the other hand, for all thermal conductivity analysis of hydrate sediments, the thermal conductivity value of sand, 0.50 W/mK was substituted. The sand would involve many other minerals and micro-pores inside and on surface. These involves would be decrease of actual thermal conductivity of sand particles. The uncertainty at boundary between sand particles and hydrate/ice layers observed in X-ray CT images is also attributed to the error of estimation of thermal conductivity. Since micro-focus X-ray CT apparatus has only $5.5 \mu\text{m}/\text{pixel}$ resolution, the actual aspect on contact between sand particles would be abundant. This abundance is strongly effective to estimate the thermal conductivity of sand particles because the heat flow can be mainly made through the contacts between sand particles.

4. Conclusions

Three-dimensional images of several natural and artificial methane-hydrate sediments were measured using a microfocus X-ray CT system. The obtained 3-D image data were applied to analyze the distribution of continuous pore in the sediment and to homogenization method. An investigation of the structural properties of sediments indicated that the channel number in sediment is a dominant factor to determine the absolute permeability. In other words, the absolute permeability of sediment increases with an increase in the ratio of horizontal-to vertical-channel densities. Furthermore, the thermal conductivity of hydrate sediments could be estimated by a homogenization method combining the 3-D X-ray CT images obtained. The estimated thermal conductivities closely agree

Table 2 Structural properties and thermal conductivity of gas hydrate sediments.

Sample	Porosity (%)		Hydrate Saturation (%)		Thermal conductivity (W/mK)	
	by 3-D CT image	by Mercury porosimeter	by 3-D CT image	by direct melting	by HM with 3-D CT image	by Hot disk
F	53.0	54.5	9.3	8.6	1.570	1.766
G	65.9	64.6	24.5	25.0	0.362	0.460
H	39.9	40.2	36.2	33.2	0.350	0.370
I	60.2	59.2	69.0	63.8	0.522	0.430

with those measured by the hot disk method. As the result, this nondestructive analysis should be useful for prediction of thermo-mechanical properties and material flow in natural gas hydrate sediments dug from permafrost areas and from oceanic sediments.

Acknowledgements

This work was funded by the Research Consortium for Methane Hydrate Resources in Japan (MH21 Research Consortium). The authors thank Miss. T. Nakao, Drs. K. Suzuki, H. Oyama, D. Katsuki and S. Takeya of AIST for useful discussions on the microfocus X-ray CT images obtained. The authors also express thank to Dr. Y. Yamamoto and Mis. M. Ohtake of AIST for variable discussions and measurements on thermal conductivity of sedimentary samples.

Notation

$\vec{x}, \vec{y}$	coordination in macro- and micro-scale elements
Ω, Y	considering subject area in macro- and micro-scales
ε	modification ratio between macro- and micro-scales
K_{ij} [W/mK]	thermal conductivity tensor in macro-scales
f [J]	the internal heat source
T [K]	temperature of subject
δT [K]	differential temperature belong to certain direction
T^0, T^1 [K]	temperature in macro- and micro-scales
Ψ	certain periodic function
ϕ^i	certain solution function of equation (5)

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要 旨

マイクロフォーカス X 線 CT を用いた MH 堆積物の浸透率および熱伝導率評価

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天然メタンハイドレート堆積物の浸透率および熱伝導率をマイクロフォーカス X 線 CT を用いて評価した。三次元 CT 像から孔隙，砂粒子，ハイドレートおよび氷を分離し，連続した孔隙ネットワーク分布（連通孔）を抽出した。実験的に得られた絶対浸透率と連通孔分布との比較から，連通孔密度の異方性が高いほど絶対浸透率が低いことが分かった。この結果は堆積物中の連通孔密度分布が絶対浸透率に大きく寄与することを示唆している。また，得られた三次元 CT 像に有限要素法の 1 つである均質化法を適用し，メタンハイドレート堆積物の熱伝導率を評価した。本手法により算出した熱伝導率と実験により得られた熱伝導率は非常に良く一致した。これらの結果から，マイクロフォーカス X 線 CT を用いた三次元 CT 像解析手法が，天然ガスハイドレート堆積物中の流体移動ならびに熱物性を算出する有効な手法であることを明らかにした。