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THE TECHNOLOGY OF SAMPLING FOR GAS HYDRATES BY HOLE BOTTOM FREEZING

Summary. The paper put forward the idea of sampling for gas hydrates by hole bottom freezing, and the method is that the temperature of the gas hydrate core is decreased on the hole bottom by cold source, the result of decreasing the temperature is the same as increasing pressure to inhibit gas hydrate dissociating and retains the core of gas hydrates. Firstly, the feasibility of hole bottom freezing method for sampling gas hydrates is analysed by research the temperature-pressure property. Secondly, it is confirmed that the dry ice is used as coolant, the alcohol as positive freezing catalyst and cooling medium by the indoor freezing experiment. Finally, the freezing sample tool is developed, the freezing loess sample is achieved. The research supplies the news ideas for the design of gas hydrates core sampler.
Key words: Gas Hydrates; Hole Bottom Freezing Sampling; Dry Ice Freezing Method; Core Sample

TECHNIKA OPRÓBOWANIA HYDRATÓW GAZOWYCH PRZY ZASTOSOWANIU ZAMRAŻANIA DNA OTWORU

Sreszczenie. Zaprezentowano ideę opróbowania hydratów gazowych przy zastosowaniu zamrażania dna otworu. Polega ona na tym, że temperatura rdzeniowanego materiału jest obniżana w dnie otworu, co wywołuje podobny efekt jak wzrost ciśnienia, uniemożliwiając dysocjację pobieranych próbek hydratów. Zaproponowano użycie suchego lodu jako czynnika chłodzącego oraz alkoholu jako katalizatora procesu zamrażania. Przedstawiono nowe możliwości projektowania rdzeniówek do poboru próbek hydratów.

1. Introduction

Gas hydrate is consists of hydrocarbon molecule trapped inside cage-like crystal structures made up of water molecules (see fig. 1), gas hydrate is also called methane hydrates or "Fiery ice" because the methane is the mainly gas molecule of gas hydrate.

Exploiting gas hydrate is based on evaluate gas hydrate, and sample drilling is the most directly method to evaluate gas hydrates. Gas hydrates are formed under the condition of low temperature (0-10°C) and high pressure (more than 10 MPa), such special condition needs high requirements on sample drilling. At present, the pressure-tight core barrel is a main truth-preserving core sampling tool, and its design mentality is that when the hydrate core (or core sample) has entered into the core chamber of the pressure-tight core barrel, the bottom of the core barrel is closed by a ball valve to enable the core to maintain its initial pressure, and a pressure compensation device is used to control the pressure in order to maintain it unchanged during the whole process of lifting the core from the hole bottom, and then a freezing preservation process is carried out after the core has been lifted to the ground. The heat preservation method of the core in the drill hole mainly uses thermal insulation materials to achieve passive thermal insulation. This method is used to inhibit hydrate decomposition by mechanically maintaining a constant pressure, the requirements for the strength of whole core, especially strength and sealing of the ball valve are quite high. Even if the sealing performance of the ball valve decreased slightly, the core would not be able to maintain the initial pressure, which may cause a failure of coring. When the design pressure of the sampler reaches a certain level, the materials and sealing performance of the sampler need to be improved a lot if the pressure is desired to be further increased, but it is not easy to be achieved.

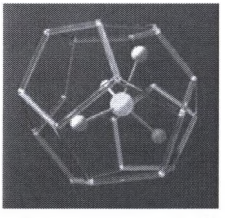

Fig. 1. Crystal structure model of hydrates
Rys. 1. Model struktury hydratu

2. Research and Practice

2.1. Feasibility analysis of freezing method for sampling gas hydrates

It can explain the theory of gas hydrates freezing sampling in quantification and ensures the optimum freezing temperature by fig. 2. The gas component of gas hydrates is that methane accounted for 87.2%, ethane accounted for 7.6%, propane accounted for 3.1%, normal butane accounted for 0.5%,isobutene accounted for 0.8%, nitrogen accounted for 0.4%, pentane accounted for 0.2%, isopentane accounted for 0.2%. From the fig. 4 we can see that critical dissociated pressure greatly decrease with the reduction of gas hydrates temperature. The dissociated dissociated pressure is 0.7 MPa when the temperature of gas hydrates is 273.2 K Supposed that dot A lies in the gas hydrates stable area, and the pressure is 8 MPa, the temperature is 283.2 K. If it make the temperature of gas hydrates reduce from 283.2 K to 253.2 K by the cold source, and the critical pressure will be reduced from 2.5 MPa to 0.3 MPa. And the gas hydrates still lies in the stable area, and its pressure is 8 MPa, it far outweighs the critical pressure of 0.3 MPa, it improves the stability of gas hydrates. If the temperature continues reducing, the critical pressure slowly decreases, so it ensures the optimum freezing temperature is 253.2 K, and is the –20°C. If the temperature of gas hydrates

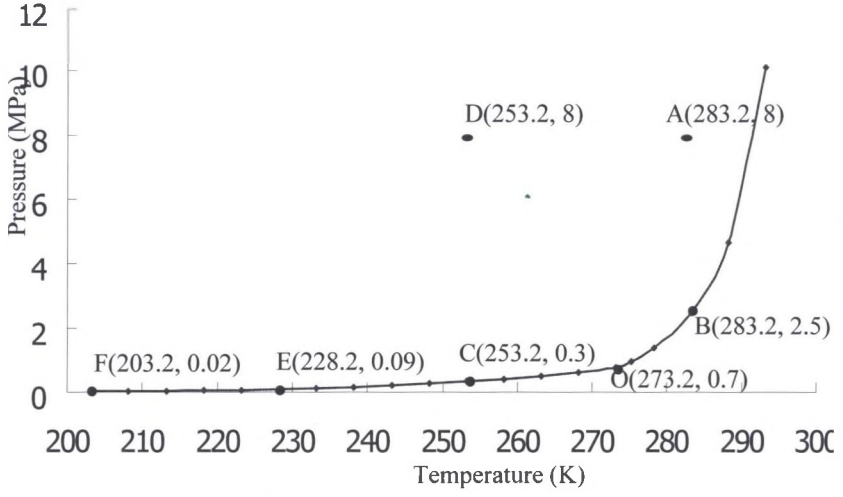

Fig. 2. The phase-change of critical pressure-temperature of typical gas hydrate
Rys. 2. Diagram przemian fazowych typowego hydratu gazowego

resuming reduce to 228.2 K, its critical pressure is only 0.09 MPa, that is to say, gas hydrates will not dissociate under the condition of normal atmosphere if maintain that temperature.

Thus, it can decrease the critical dissociated pressure if reduce the temperature of core in the course of sampling, and the design difficulty of pressure-tight mechanism for truth-preserving core sampling of gas hydrates is reduced greatly; even it is possible to achieve the truth-preserving core without pressure-tight mechanism indeed.

2.2. Indoor freezing simulate experiment

Choosing the optimum cold source is key to gas hydrates freezing sampling, that is to say, choosing the power cold source and reasonable freezing ways, and it can be applied in design freezing core sampling. So it is available to choose the dry ice is used as coolant, the alcohol as positive freezing catalyst and cooling medium by freezing experiment.

According to the thermophysical property of gas hydrates, choosing the sediment contains the ice as simulate sampler. The sand of ice content 15% simulate sample is used to freezing experiment.

The experiment device is consisting of preserve cold energy structure, freezing structure (see fig. 3).

The preserve cold energy structure can preserve the cold energy of dry ice, the special sublimation freezing structure and material is chose for improve the retain time of dry ice. The freezing structure can freeze the simulate sampler, the copper pipe is chose the material of sampler pipe in order to improve the efficiency of heat transfer. The dry ice is filling in the special sublimation freezing structure and material, the simulate sampler place in the copper sampler pipe. It fill with the alcohol from the upper the device, the alcohol turn the low temperature alcohol after heat transfer with dry

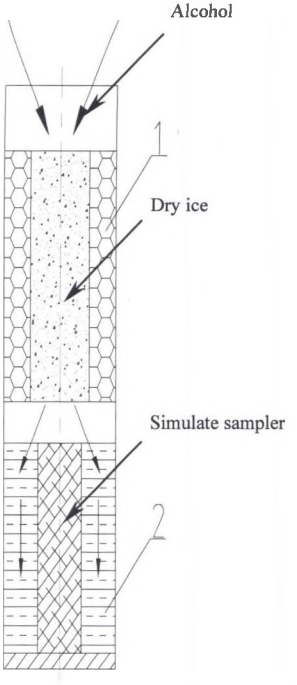

Fig. 3. The freezing experiment device:
1 - preserve cold energy structure
2 - freezing structure

Rys. 3. Urządzenie zamrażające:
1 - izolacja termiczna; 2 - element zamrażający

ice, and the low temperature alcohol freezing simulate sampler. The result of experiment is seen in fig. 4.

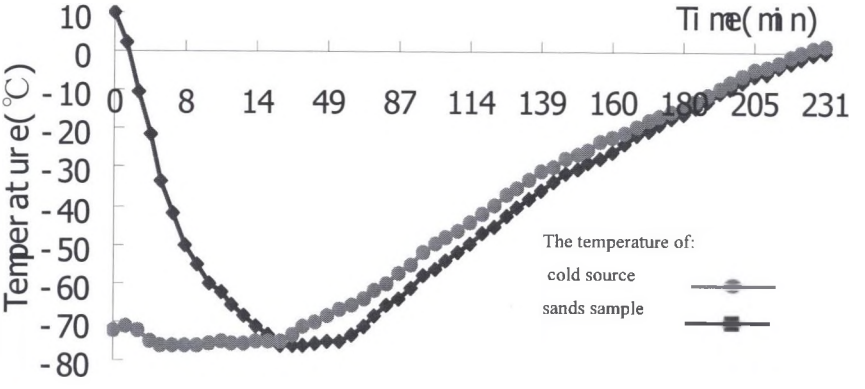

Fig. 4. The sands temperature variations of freezing experiment
Rys. 4. Zmiany temperatury piasków podczas eksperymentu

The sample temperature is decreased to the optimum freezing of -20°C in a short time by the ways of dry ice freezing, and the sample average temperature decreasing speed is reach to 8-11°C /min, and it can be use to design the sampling core, thus, the dry ice freezing method is decided to the optimum freezing method.

2.3. The hole bottom freezing core sample for gas hydrate

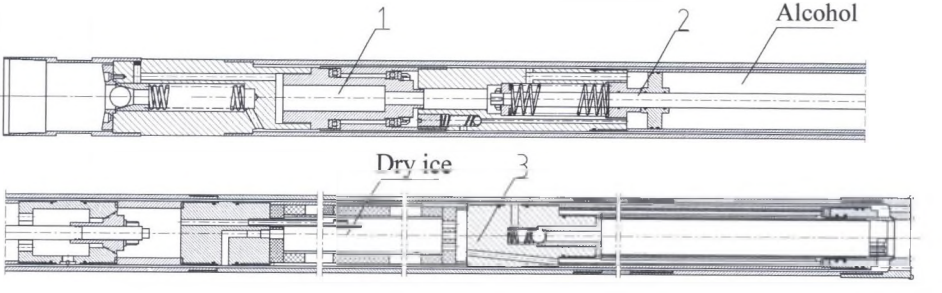

Fig. 5. The structure of hole bottom freezing core sample for gas hydrate
Rys. 5. Budowa próbnika

The prototype of dry ice hole bottom freezing core sample tool is developed. According to the conclusion of simulate freezing experiment, the dry ice used as coolant, the alcohol as

positive freezing catalyst and cooling medium, design the gas hydrate freezing core sample. The tool is making up of single action mechanism, control mechanism and freezing mechanism. (see fig. 5) Single action mechanism can make the inner and outer pipe assembly independent rotary to avoid disturbance core. The control mechanism achieves alcohol enter into dry ice and accelerate the dry ice sublimation by the way of putting steel ball into the tool to change the direction of drilling fluid circulation. Freezing mechanism can preserve dry ice in the bottom hole, which is copper as the material of core barrel makes the core rapid freeze by the cold source to achieve freezing core in the bottom of hole.

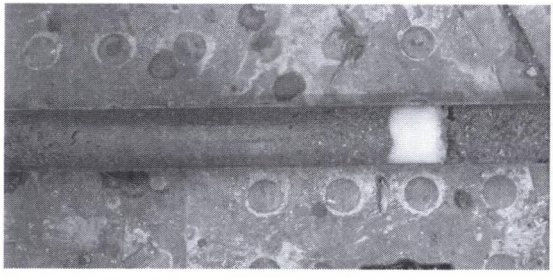

Fig. 6. The freezing sands core sample
Rys. 6. Próbka zamrożonych piasków

In order to inspect the function of the sample tool, the freezing sample experiment is done in the sands stratum, the result of experiment proves that the sample tool holds reliable in the process of drilling. And the freezing sands sample is achieved, especial the water sample freeze into the ice sample (see fig. 6). It further prove that the freezing sample tool can freeze the core in the bottom of hole.

3. Conclusions

1) The sample drilling is the most directly method to evaluate gas hydrates, the feasibility of freezing method for sampling gas hydrates is analyzed by Observational and quantitative method analysis the quality of temperature and pressure of gas hydrate, the method is to decrease the critical dissociated pressure of gas hydrate and inhibit gas hydrate dissociating by means of reducing core temperature. The optimum freezing temperature is -20°C. it is possible to achieve the truth-preserving core without pressure-tight mechanism indeed.

2) The dry ice freezing ways is that the dry ice is used as coolant, the alcohol as positive freezing catalyst and cooling medium. The temperature dropping speed of sample arrives at 8-11°C /min due to the power refrigerate capacity of dry ice. It is available to apply dry ice freezing method in the design of sample tool for freezing sample of gas hydrates in the well bottom.

3) According to the conclusion of simulate freezing experiment, design the gas hydrate hole bottom freezing core sample. It prove that the technology of hole bottom freezing is feasibility by the freezing drilling experiment, it can achieve freezing core in the bottom of hole, it can be applied in the design of the truth-preserve core barrel for gas hydrate, and it supplies the news ideas for the design of gas hydrates core sampler.

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