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#### ABSTRACT

The analysis of core samples is an essential component in the evaluation of natural gas hydrate (NGH) reserves. To prevent NGH volatilization in the sampling process, the commonly employed pressure-temperature preservation technique is used to maintain the temperature-pressure condition of the NGH core at its in situ value. However, the structure of a typical pressure-temperature preservation sampler is notably complicated, the diameter of the core is small, and the insulation effect is poor. To solve these problems, we propose a hole-bottom freezing sampling technique that uses liquid nitrogen as a cold source to decrease the temperature of the NGH core, which reduces its critical breakdown pressure and promotes the self-preservation of NGHs. We also propose two different types of hole-bottom freezing samplers, which are denoted as the cold-source built-in freezing sampler and the cold-source external freezing sampler. Both experimental tests and numerical simulation of the heat-transfer process during core freezing were conducted to evaluate the freezing efficiency of the new method. We calculated and compared the liquid-nitrogen consumption of the two different types of samplers in the process of NGH sampling. The results demonstrate that greater efficiency is obtained using the proposed cold-source external freezing sampler.

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### 1. Introduction

World-wide natural gas hydrate (NGH) reserves are a notably large potential energy resource, estimated at over  $15 \times 10^{12}$  tons of oil equivalent. In fact, utilization of only 17%–20% of this resource would be sufficient to supply the world's energy requirements for 200 years (Makogon, 2010). NGHs are stable at low temperatures and high pressures and are usually located in permafrost regions, deepwater seas, and lake sediments (Makogon et al., 2007; Fereidounpour and Vatani, 2014). The analysis of core samples is an essential component in the evaluation of NGH reserves. However, when the temperature rises or the pressure drops, NGH will decompose; therefore, the drilling of core samples is a difficult task due to the volatility of the NGHs. Currently, the primary methods used for NGH core sampling include pressure-preservation and temperature-preservation techniques.

The NGH pressure-preservation core-sampling technique seeks

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to maintain a core's in situ pressure after completion of the sampling process. To this end, the technique uses a high-pressure ball valve or a flapper valve at the bottom of the pressure barrel, operated by a torsional spring, to seal the pipe once the sampling is completed. A pressure accumulator cylinder filled with nitrogen gas at high pressure resides outside of the sampling pipe. A pneumathode is contained inside the pressure-accumulator cylinder that is connected to a piston. The pressure barrel and the pressure pipe are linked together with a high-pressure hose. When the pressure inside the barrel decreases, the high-pressure nitrogen gas in the pressure-accumulator cylinder forces the piston to move down through the compensating hole to maintain the in-situ pressure of the pressure pipe. The high pressure inside the barrel prevents the volatilization of the NGHs core during sampling (Zhu et al., 2011). A schematic of the NGH pressure-preservation coresampling technique is shown in Fig. 1. The NGH pressurepreservation systems currently in use include the pressure core barrels (PCB) used in the Deep Sea Drilling Project (DSDP) (Peterson, 1984), pressure core samplers (PCS) used in the Ocean Drilling Program (ODP) (Dickens et al., 2000; Milkov et al., 2004), FUGRO pressure corers (FPC), and hydrate autoclave coring equipment (HYACE) rotary corers (HRC) developed under the EU-





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Fig. 1. Schematic of the NGH pressure-preservation core-sampling technique.

sponsored HYACINTH program (Schultheiss et al., 2009). However, suppression of hydrate dissociation by maintaining a core's in situ pressure requires a high-strength sampler and a high-integrity mechanical seal. If the sealing property of the mechanical seal structure is at all compromised, then the core's in situ pressure will not be preserved, resulting in coring failure. Finally, note that due to the ball-valve structure, these samplers can only provide a small diameter core.

The NGH temperature-preservation core-sampling techniques include both passive and active schemes. A passive temperaturepreservation technique includes a sampling tube, a pressure barrel, and an outer pipe. The air within the segment between the pressure barrel and the outer pipe is evacuated and filled with heatinsulating materials. In addition, the surfaces of the pressure barrel and sampling tube are plated with insulating materials. Implementing these measures can substantially reduce heat transmission, as shown in Fig. 2(a). An active temperature-preservation technique decreases the temperature of NGH cores through the use of liquid ammonia or thermoelectric refrigeration. The liquidammonia approach injects liquid ammonia into the annular gap created between the sampling pipe and the outer pipe, and then the temperature of the NGH core is reduced by heat absorption due to liquid-ammonia gasification. The thermoelectric-refrigeration technique uses a lithium-ion battery to decrease the temperature of the core. The lithium-ion battery is installed at the top of the sampling pipe, as shown in Fig. 2(b). The air within the segment between the sampling pipe and the outer pipe is then evacuated and filled with heat-insulating materials (Zhu et al., 2011). The temperature-preservation technique is always used together with the pressure-preservation technique, which improves sampler reliability. The pressure and temperature core sampler (PTCS) (Wakishima et al., 1998; Kawasaki et al., 2006), the multiple autoclave corer (MAC), and the dynamic autoclave piston corer (DAPC) used in the RV Sonne cruises are of this type (Bohrmann et al., 2007; Heeschen et al., 2007; Abegg et al., 2008). The passive temperature-preservation technique is simple, inexpensive, and commonly used, whereas the active temperature-preservation techniques provide improved core temperature maintenance. However, the structure of the active temperature-preservation technique is more complex than its passive counterpart and is also difficult to realize, given the strict size limitations for the sampler.

Therefore, we propose a new hole-bottom freezing technique to avoid these problems. The technique uses liquid nitrogen as a cold source to reduce the temperature of the NGH core to prevent decomposition. In this paper, the NGH hole-bottom freezing technique is introduced, and two different types of hole-bottom freezing samplers are proposed. Based on the freezing core experiments and the heat-transfer simulations of the core freezing process, the hole-bottom freezing technique is shown to be feasible. However, liquid nitrogen is easily gasified, which reduces the efficiency of the process. We calculated and compared the liquidnitrogen consumption of the two different types of core-freezing samplers proposed to determine the optimal structure.

#### 2. NGH hole-bottom freezing technique

The hole-bottom freezing technique does not have the limitations associated with the pressure-preservation and temperature-preservation techniques. The hole-bottom freezing technique utilizes a sampling pipe and an outer pipe. Liquid nitrogen is injected into the annular space between the sampling pipe and the outer pipe to reduce the NGH core to subzero temperatures in the sampling process. The low temperature reduces the critical NGH breakdown pressure of the core and promotes the self-preservation of NGHs and also prevents volatilization during sampling (Yakushev and Istomin, 1992). Two different types of hole-bottom core freezing samplers were designed, which are denoted as the cold-source built-in freezing sampler and the cold-source external freezing sampler.

The structure of the cold-source built-in freezing sampler is illustrated in Fig. 3, and its operation is shown in Fig. 4. During the drilling process (Fig. 4(a)), the liquid nitrogen chamber is full and is sealed using a vacuum heat-preservation chamber, which can substantially reduce heat transmission. The liquid-nitrogen chamber valve prevents liquid-nitrogen flow into the freezing chamber. When drilling is completed, lifting the drill forces the male spline to move downward. The male spline blocks the mud channel, which leads to an increase in the mud pressure that forces the piston at the top of the mud chamber to open, thereby allowing mud to flow from the mud inlet, through the mud outlet, and finally into the mud chamber on the upside of the control piston. The pressure of the mud chamber pushes the control piston downward to increase the internal pressure of the liquid-nitrogen chamber. The liquid-nitrogen chamber valve is forced downward under the internal pressure of the liquidnitrogen chamber, and then the liquid nitrogen is injected into the freezing chamber through the liquid-nitrogen channel. The temperature of the NGH core in the core chamber is decreased by heat absorption due to liquid-nitrogen gasification in the freezing chamber in addition to heat exchange with the liquid nitrogen and evolving cold nitrogen gas (Fig. 4(b)). The nitrogen gas is discharged through the nitrogen-gas outlet. In the process of freezing the core, the heat-insulation pipe maintains the temperature of the liquid nitrogen in the freezing chamber (Fig. 4(c)).

We propose a design for the cold-source external freezing sampler that holds the liquid nitrogen in a fisher. The sampler structure therefore includes a fisher and drilling tool, as shown in Fig. 5. The operation of the cold-source external freezing sampler is as shown in Fig. 6. In the process of drilling (Fig. 6(a)), liquid nitrogen is stored in the fisher's vacuum-insulated heat-preservation chamber, rather than the drilling tool (as in a cold-source built-in freezing

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